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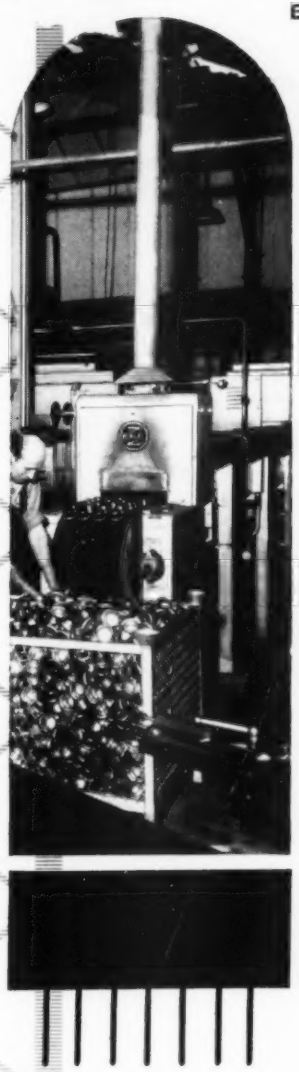

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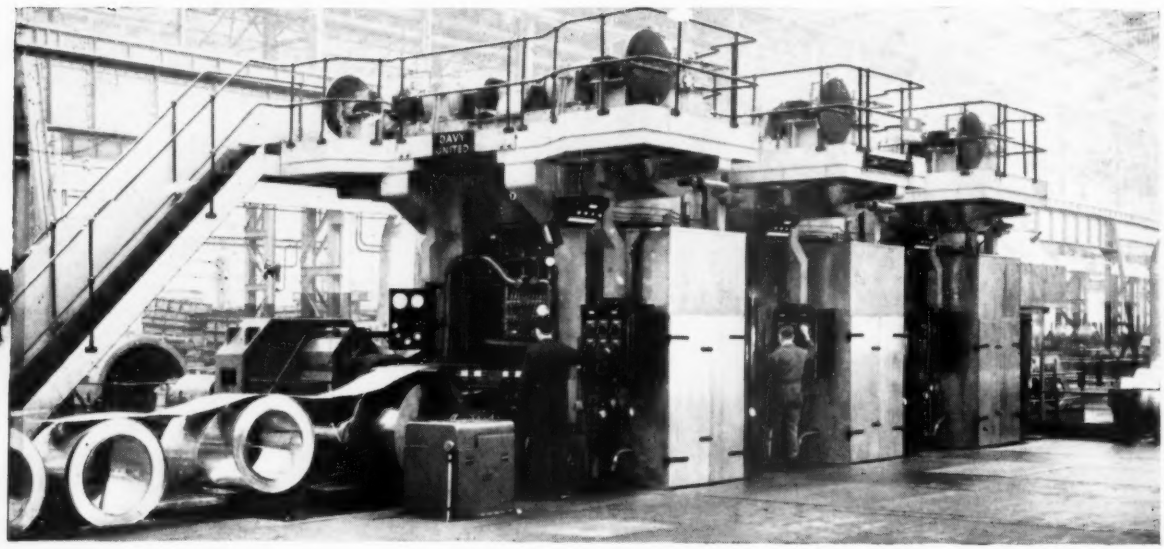

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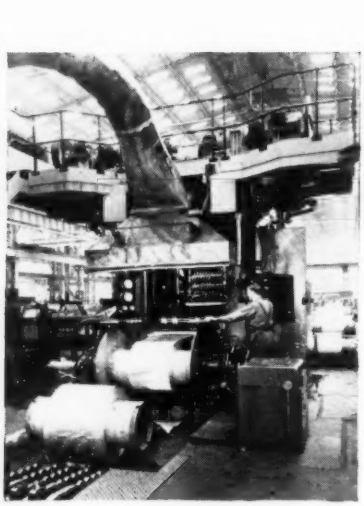

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INCORPORATING THE METALLURGICAL ENGINEER

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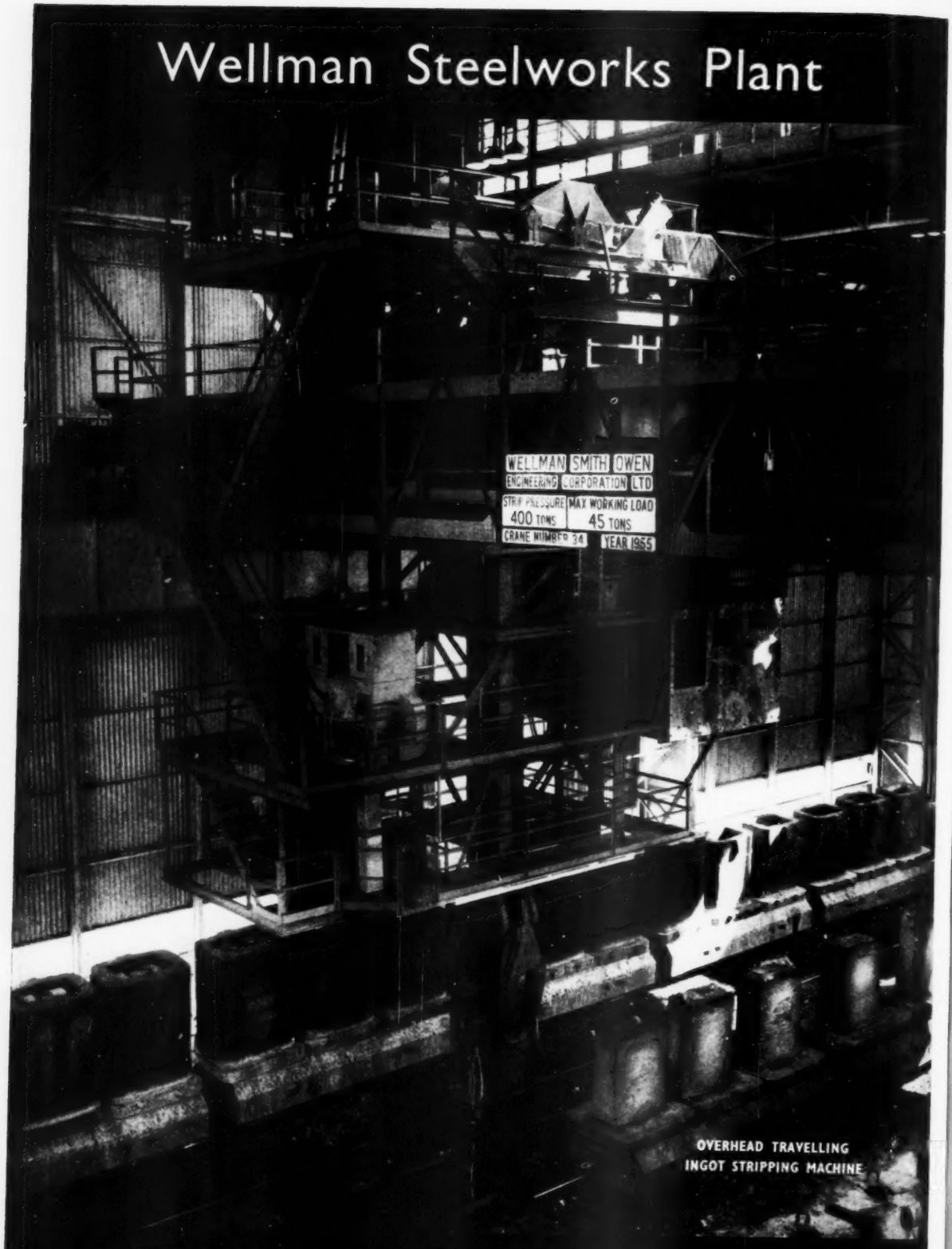

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THE BRITISH JOURNAL OF METALS

INCORPORATING THE "METALLURGICAL ENGINEER,"

NOVEMBER, 1958 Vol. LVIII. No. 349

Geneva Survey

THE second International Conference on the Peaceful Uses of Atomic Energy was held, like the first in 1955, in Geneva, and in the course of an evening lecture, Sir John Cockcroft, O.M., Member of Scientific Research, United Kingdom Atomic Energy Authority, surveyed the work of the Conference, and outlined the likely course of the peaceful development of atomic energy in the world.*

The three years since the previous conference have been notable for the coming into operation of the world's first large scale nuclear power stations at Calder Hall, Shippingport and recently Siberia. First impressions have been that these stations have been docile and well-behaved. They can generate electricity for months on end until some minor fault develops; the most usual faults have been the faults of conventional components which require the normal amount of maintenance. One of the surprising, but nevertheless gratifying, features has been the small number of defective fuel elements, due in no small measure to the quality of the sheathing. The power stations so far built have been either dual purpose stations or demonstration stations, and would not be economic as commercial power stations. The experience in their operation has, however, been invaluable in preparing the way for the next generation, which in most cases will be fully commercial nuclear power stations with credits for plutonium based on its real value for civil purposes.

Three main types of second generation power stations were described at the Conference: graphite moderated gas cooled reactors; pressurised and boiling water reactors; and heavy water moderated reactors. Although much reduced below those of Calder Hall and Shippingport, the capital costs per kilowatt of the first of the commercial stations are still more than twice those of coal or oil fired stations, but they are likely to fall appreciably during the next decade as output goes up from 300 MW. to 500 MW., and as a result of straightforward engineering improvements. The boiling water reactor power stations seem to be growing in favour as a result of good performance of the reactor experiments, and as a result of their low system pressure and comparative simplicity, they may achieve very low capital costs in the next five years.

Fuel costs are the second important component of overall costs, and range from 20% to 40% of the unit cost, depending on whether natural uranium or near-natural uranium or more highly enriched fuel is used. The graphite moderated and heavy water moderated reactors will have the lowest fuel costs, but even the light water moderated reactor fuel costs will be lower than conventional fuel costs.

High load factors are essential to counteract the present high capital costs, and in the case of the first group of stations this should be readily achieved. Even on a 75% load factor basis, the 500 MW. stations due to be completed in 1962 is forecast to achieve parity with coal fired stations in areas in Britain away from coalfields. Futhermore fuel costs are likely to fall as burn-up increases and as uranium prices fall.

By the late 1960's, as the installed capacity of nuclear power stations increases, the available load factor will fall, but this is more than likely to be compensated by further reduction of capital costs resulting from higher temperatures of operation and higher ratings associated with a switch to ceramic fuels. Nuclear power costs in Britain are therefore forecast to fall well below conventional costs by the late 1960's. The date of achieving parity will be later in countries such as the United States, where hydroelectric stations and stations using low cost coal or natural gas can generate power very cheaply. India has reported that the first 150 MW. power station to be built could achieve parity. The date for achieving parity ranges from 1963 to 1973, depending on the circumstances of individual countries. The date will determine the rate at which large scale installations of nuclear power stations will develop.

By the late 1960's new, third generation, types of stations may be coming into service. The organic liquid moderated reactor shows promise with its low system pressure and its non-corrosive non-radioactive coolant, and progress has been made at Oakridge in overcoming the difficult compatibility problems of the aqueous homogeneous reactor, although the stability of the fluid fuel is still a crucial point. The sodium graphite reactor experiment will provide important information on sodium technology and on metallic fuel elements operating at high temperatures, and the high temperature gas cooled reactor projects using all-ceramic fuels may well be important for propulsion as well as for land use by the end of the 1960's.

A number of interesting papers were presented on fuel cycles, and it appears that, with the general trend to the use of uranium oxide fuels, most reactors built after 1965, with the possible exception of the heavy water reactors, will require some enrichment, which may be provided by U-235 from diffusion plants or by using plutonium from earlier reactors. Thereafter the reactors could operate on a natural or slightly enriched uranium feed by recycling plutonium and using it in the oxide form mixed with uranium oxide. Another possible fuel cycle is to feed plutonium to reactors of the fast breeder type which will have a positive gain factor for plutonium. Fast reactors depend on the availability of plutonium from thermal reactors and are unlikely to contribute much to world power before the 1970's. Thorium as a fuel for thermal reactors may well be coming into use by the late 1960's, especially since large supplies will be available as a by-product of uranium mining.

* Printed in the November issue of *Atom*, the monthly information bulletin of U.K.A.E.A.

Meeting Diary

11th November

Incorporated Plant Engineers, Manchester Branch. "Radio-isotopes in Industry," by W. G. Busbridge. Engineers' Club, Albert Square, Manchester. 7.15 p.m.

Institution of Mechanical Engineers. "Fatigue Testing of Vehicle Components", by T. C. F. Scott. 1, Birdcage Walk, Westminster, London, S.W.1. 6 p.m.

Sheffield Metallurgical Association. "Brick Quality and Furnace Construction Methods, their Effects on Refractories Life in Steel Plants," by G. A. Smith. B.I.S.R.A., Hoyle Street, Sheffield. 7 p.m.

12th November

Institute of Fuel, North Western Section (Merseyside Sub-Section). "Calder Hall Plant—Principles and Operating Experiences," by J. Gawthrop. **Joint Meeting with the Liverpool Engineering Society.** 9, The Temple, Dale Street, Liverpool. 6 p.m.

Liverpool Metallurgical Society. "The Gas Cutting of Metal Plates," by Dr. L. C. Bannister. **Joint Meeting with the Liverpool Branch of the Institute of Welding.** Picton Library, 7 p.m.

Sheffield Society of Engineers and Metallurgists. "The World's Largest Walking Dragline," by C. McL. Cameron. **Joint Meeting with the Yorkshire Branch of the Institution of Mechanical Engineers.** The University Building, St. George's Square, Sheffield. 6.30 p.m.

Society of Chemical Industry, Corrosion Group. "Plastics and other Polymeric Materials as Sources of Corrosive Vapours," by H. G. Cole and Mrs. V. E. Rance; "Plastics for Protection against Corrosion," by A. C. Docherty and H. Hughes. 14, Belgrave Square, London, S.W.1. 6.30 p.m.

13th November

Incorporated Plant Engineers, Glasgow Branch. "Packaged Oil Fired Boilers," by A. Wilson. Scottish Building Centre, 424/427, Sauchiehall Street, Glasgow. 7.15 p.m.

Incorporated Plant Engineers, North East Branch. "The Gas Turbine," by H. R. Beaton. Roadway House, Oxford Street, Newcastle-upon-Tyne. 7 p.m.

14th November

Manchester Association of Engineers. "The Machining of Large Light Alloy Components by Computer Control," by J. Gregson and A. Flett. Engineers' Club, Albert Square Manchester. 6.45 p.m.

West of Scotland Iron and Steel Institute. "Slag Composition and Influence on Blast Furnace Practice", by K. C. Sharp. 39, Elmbank Crescent, Glasgow. 6.45 p.m.

17th November

Institute of Welding, Manchester and District Branch. "Design for Welding." A Panel of Experts will answer questions. Engineers' Club, Albert Square, Manchester. 7.15 p.m.

Sheffield Society of Engineers and Metallurgists. "Progress in Rod and Bar Rolling," by R. Stewertson. The University Building, St. George's Square, Sheffield. 7.30 p.m.

18th November

Sheffield Metallurgical Association. "Vacuum Melting," by G. L. Willan. B.I.S.R.A., Hoyle Street, Sheffield. 7 p.m.

19th November

Incorporated Plant Engineers, Kent Branch. "Cathodic Protection," by L. B. Hogben, O.B.E. Railway Hotel, Dartford 7 p.m.

Institute of Metal Finishing, London Branch. One-Day Symposium on "Technical Control in Electroplating." 10.45 a.m. to 12.45 p.m.—"Chemical Analysis of Plating Solutions," by K. E. Langford, and "Control of Electro-deposition Processes by Examination of Deposits," by T. E. Such; 2.15 p.m. to 4 p.m. —"Trouble Shooting in the Plating Shop," by C. F. Corfe, and "Requirements and Inspection of Metallic Finishes for Service Use" by D. W. Smith. Charing Cross Hotel, Strand, London. Applications to Mr. S.W. Baier, 9E, Cleveland Road, London, W.13.

Manchester Metallurgical Society. "Vacuum Melting," by H. C. Child. Manchester Room, Central Library, Manchester. 6.30 p.m.

20th November

North-East Metallurgical Society. "Gemstones," by T. Jones. Members are invited to bring their ladies to this meeting. Cleveland Scientific and Technical Institution, Middlesbrough. 7.15 p.m.

25th November

Incorporated Plant Engineers, South Wales Branch. "Corrosion," by D. W. Marshall. South Wales Engineers' Institute, Park Place, Cardiff. 7.30 p.m.

Sheffield Metallurgical Association. "Development in the Use of the Absorptiometer and Spectrophotometer for Methods of Analysis," by A. A. R. Wood. B.I.S.R.A., Hoyle Street, Sheffield. 7 p.m.

27th November

Institution of Engineering Inspection, North Western Branch. "Service Failures in Engineering," by H. Hutchings. Engineers' Club, Albert Square, Manchester. 7.30 p.m.

28th November

Manchester Association of Engineers. "Electro-Mechanical Themes in Machine Tool Design," by The Rt. Hon. The Earl of Halsbury. Manchester College of Science and Technology, Manchester. 7.30 p.m.

2nd December

East Midlands Metallurgical Society. Film: "Refining Nickel." H. C. Castell. Electricity Showrooms, Smithy Row, Nottingham. 7.30 p.m.

Sheffield Metallurgical Association. "Steelmaking in Small Furnaces," by T. A. Cosh. B.I.S.R.A., Hoyle Street, Sheffield. 7 p.m.

Society of Chemical Industry, Corrosion Group. Discussion of Papers Recently Published by Members of the Group. 14, Belgrave Square, London, S.W.1. 6.30 p.m.

3rd December

Institute of Welding, Manchester and District Branch. "Welding in the Rocket Motor Industry," by F. J. Wilkinson. Reynolds Hall, College of Science and Technology, Manchester. 7.15 p.m.

Manchester Metallurgical Society. "Metallurgical Control in Mass Production," by J. E. James. Manchester Room, Central Library, Manchester. 6.30 p.m.

4th December

Institute of Fuel, North Western Section (Merseyside Sub-Section). "The Use of Additives to Reduce Boiler Fouling and to Prevent Low Temperature Corrosion," by W. D. Jarvis Liverpool Engineering Society's Rooms, 9, The Temple, Dale Street, Liverpool. 7 p.m.

5th December

Society of Chemical Industry, Manchester Section. Jubilee Memorial Lecture: "The 'Conveyor' Concept of Water in Industry," by E. L. Streatfield. Robinson Lecture Theatre, The University, Manchester, 6.30 p.m.

9th December

Sheffield Metallurgical Association. "On Getting to Know Ceramic Technology," by Dr. N. F. A. Astbury. **Joint Meeting with the Sheffield Branch of the Institute of Clay Technology.** B.I.S.R.A., Hoyle Street, Sheffield. 7 p.m.

Society of Instrument Technology, Manchester Section. "An Exercise in Instrumentation," Manchester Room, Central Library, Manchester. 6.30 p.m.

11th December

Liverpool Metallurgical Society. "The Metallurgical Applications of High Resolution Electron Microscopy," by Dr. J. Nutting. Library, Dept. of Metallurgy, The University of Liverpool. 146, Brownlow Hill, Liverpool, 3. 7 p.m.

North East Metallurgical Society. "Bearing Metals from the User's Viewpoint," by R. J. Brown. Cleveland Scientific and Technical Institution, Middlesbrough. 7.15 p.m.

Pressure Vessels and the Problem of Brittle Fracture

By H. Harris, B.Sc., Ph.D.

Although the mechanism of brittle fracture at low stresses is by no means fully understood, considerable progress has been made as a result of investigations carried out in recent years. In his recent Presidential Address to the West of Scotland Iron and Steel Institute, which is reported here, the author discussed the present status and showed that an enlightened outlook can now be achieved.

OPINIONS may vary concerning what the problem of brittle fracture really is, so that it is preferable to have as clearly in mind as possible the problem as seen by the manufacturer and user of pressure vessels. Engineers are concerned about the fact that important welded structures have failed by the initiation and rapid propagation of fractures *at low applied stresses*. In such instances the fractures have been of a brittle character, even though the steel would have been expected by conventional standards to have behaved in a ductile manner. The real problem was not that the steel failed in a brittle manner, but that the failures were under conditions of applied stress when a failure, even of small dimensions, was not expected, and certainly catastrophic collapse or rupture was not even dreamt of. Such failures have occurred in a ship lying calmly at its berth, and in a storage tank being filled with water prior to hydrostatic test. This behaviour, of particular significance to the merchant navies during the late war, was sufficiently frequent and alarming that considerable efforts have been made to investigate the fundamental facts. In spite of these efforts, which are of international scope, the problem is by no means fully understood. Sufficient research has, however, been carried out to lead to a reasonable understanding of the problem, and to form the basis for an intelligent appreciation of what can and what should not be done in the design, manufacture and use of pressure vessels.

The purpose of this address is not to deal in a chronological order with the work that has been published, but rather to take the present status and to show that an enlightened outlook can now be achieved. Let us, therefore, begin with a description of two major contributions that have originated in the British Isles. In the first place there is the work of Robertson at the Naval Construction Research Establishment at Rosyth. Robertson has shown that when a crack is initiated in a sample of steel held under a constant tensile stress, but with a temperature gradient so chosen that the crack is initiated at a low temperature, the crack is arrested at some higher temperature, called the crack arrest temperature. It is perhaps somewhat erroneous to define this temperature as the point where the crack stops. Robertson actually uses the temperature where the crack begins to show the change from cleavage to shear. This temperature is dependent upon the stress applied, so that in plotting this dependency we achieve a Robertson curve of the type shown in Fig. 1. From this figure it will be seen that for the particular steel there is a transition temperature, of the order of 0° C., above which the crack will not propagate at low stress levels, but below which a crack, once initiated, will continue to travel even though the applied stress is only of the order of 4 tons/sq. in.

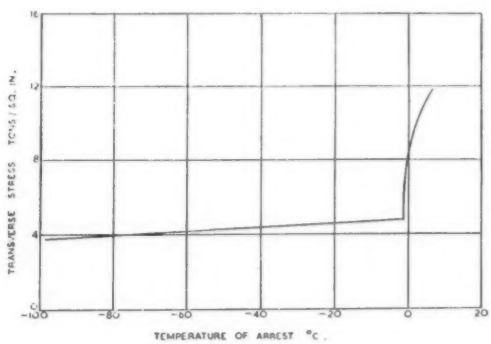


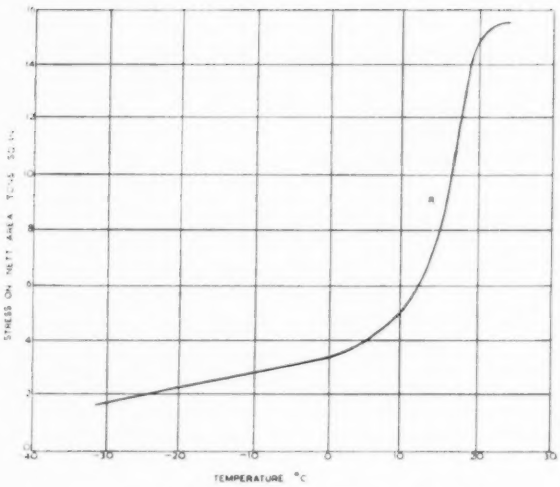
Fig. 1.—Robertson test results.

It will be appreciated how the engineer and the metallurgist welcomed such a clear exposition of one facet of the brittle fracture problem. The Robertson curve, however, is not all-sufficient. The experiment is artificial in that the method of crack initiation is quite removed from behaviours in practice; the data relates primarily to the propagation of an initiated crack.

Following this important work at the Naval Construction Research Establishment, Wells and his collaborators at the Abington Laboratories of the British Welding Research Association were exploring a method of test much more representative of actual happenings. In the work at B.W.R.A., welded test plates 1 in. in thickness and 3 ft. in width were made with artificial defects in the weld. The defect took the form of a fine saw-cut extending a very restricted distance into the parent metal. The welded plate was cooled to a pre-selected temperature and then stressed uniformly in the direction of the weld. Using an ordinary mild steel, it was found that a relationship of the type shown in Fig. 2 existed between the temperature and the stress necessary, in this instance, to initiate failure. Again it will be seen that some type of transition behaviour exists.

From a consideration of these experiments it will be appreciated that conditions very similar to actual failures are being produced in the laboratory, and that as a consequence a tool of supreme importance appears to have been forged and should be exploited.

In view of the necessity to make progress as rapidly as possible with the limited resources available, the Research



ig. 2.—Wide plate test results for mild steel.

Laboratories of Colvilles, Ltd., undertook an investigation to determine the influence of residual stresses. As a result of this series of tests it was shown by Kennedy that the presence of the residual stresses consequent on the welding operation were necessary if fracture at stresses lower than the yield point were to be initiated. This was in fact an achievement of real merit and significance. Whatever opinion a person might have harboured privately, there was a tremendous body of informed opinion that held to the belief that residual stresses had played no part in the brittle fracture failures. Now at last that opinion is shattered, and it is now recognized that stress relief has a decisive effect on whether a structure will or will not fail in a brittle manner at low levels of applied stress.

It might appear that at this point those interested in pressure vessels could afford to sit back and say that so far as they were concerned the problem was settled—apply stress relief and the fear of brittle fracture vanishes. However much truth there would be in such an attitude, it is not the complete story: the problem of brittle fractures during construction and in vessels that cannot be stress-relieved would remain.

It happened that Fig. 2 had been obtained from specimens which had been welded using rutile-coated electrodes. The choice of such electrodes was not altogether fortuitous, as they were at that time being widely used for the site construction of storage tanks on account of the facility with which they could be used by the welders. It had also been thought that, because of the good notch impact properties, they would be a reliable choice when the possibility of brittle fracture was taken into account.

A realization of how misleading conclusions can be when made from some hypothesis, which, though widely held, was nevertheless unsupported by practical data, became evident when the wide plate tests were repeated on specimens the root runs of which were deposited using low-hydrogen electrodes. Fig. 3 shows the two curves, and a significant difference is clearly to be seen.

It has been stated earlier that these experiments by Wells at the B.W.R.A. Laboratories reproduce actual conditions that are encountered in practice. An unbiased assessment will, it is believed, allow that the actual conditions of the test are what one could expect in a construction involving butt welds. The only proviso would possibly be that the severity of the initiating defect in Wells' test specimens is much more severe than would be found in even reasonably good industrial practice. Also, the results of the tests undeniably reproduce what has been found in brittle fracture failures, namely, fractures in welded constructions initiated at low applied loads. It is therefore difficult to believe that these experiments cannot be translated directly into practical terms. Arising from this is the conclusion that if a structure, as such, has a transition temperature, and if this transition temperature, as shown by the Wells' data, has real significance, it cannot at present be derived on the basis of the conventional laboratory determinations of notch impact properties of the materials, for example, plate or weld metals, employed in the construction.

In these circumstances it is not strange to find that, apart from a somewhat general appreciation that the lower the transition temperature as determined, for example, by Izod or Charpy tests, the more liable is a steel to brittle fracture, no satisfactory correlation between brittle fracture failures and such properties has been established. This condition persists in spite of the tremendous efforts that are known to have been expended in trying to find some such correlation.

It seems clear that the liability of a structure to brittle failure can only be assessed on the basis of tests in which both the parent and the weld metals participate. If for no other reason than this, the future exploitation of such tests as those of Wells in Great Britain and of Pellini in the United States will be awaited with considerable interest.

One aspect of the observation of Wells has, however, been neglected in this discussion, namely the "severity" of the failure in his tests. Whilst in Fig. 3 the relationship between stress and temperature for crack initiation is plotted, no account is taken of the severity of the cracks, assessed in the experiments by their length. With the varied experimental conditions, differences in this severity were noted; some cracks traversed the full width (3 ft.) of the specimens, whereas others were only of a few inches in length. It is in this respect that a suggestion of Rankin from Colvilles' Research Laboratories proved particularly fruitful. When the Robertson

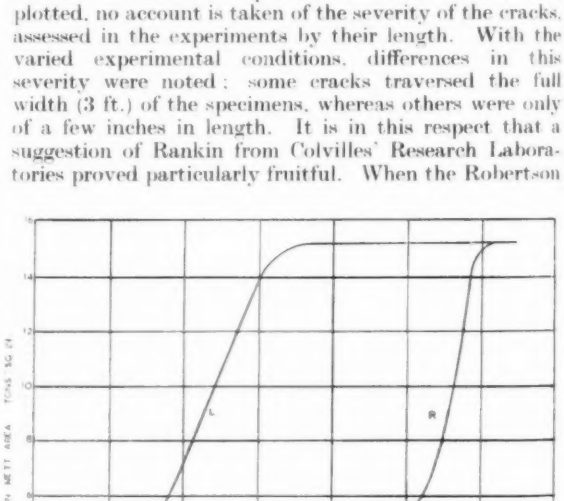


Fig. 3.—Wide plate test results for mild steel.

curve is added to those of Fig. 3, as has been done in Fig. 4, a really comprehensive picture of the brittle fracture problem becomes evident. From the Wells curves we have the temperature and stress conditions under which a crack is initiated from the artificial defect, and dependent on the relationship between this point and the Robertson curve will be the progress of the crack. If the point is to the left and above the Robertson curve, the crack will not be arrested—the failure will be complete. If the point is to the right and below the Robertson curve, the crack will not propagate indefinitely but will be stopped in the plate material. This is entirely an attractive picture of the brittle fracture problem and only further work will show whether, although the basic facts are correct, the interpretation has been over-simplified.

It must be realized that whilst the Robertson curves for plates are made under closely controlled conditions, and whilst the assessment of the experimental results is reasonably precise, at least for plates which are not particularly thick, such precision is not attained in the Wells tests because the initiating defect, as it exists immediately prior to fracture cannot, by the very nature of things, be other than variable. It is not unexpected, therefore, that the scatter on the Wells data is appreciable. This lack of precision is however not unknown, and not necessarily greater than in other studies of transition behaviours, and in the wide plate tests seems to be resolvable only by increasing the number of tests so as to establish the resultant curves with reasonable accuracy.

One of the many problems remaining concerns the reason why the electrodes used in these experiments play such a large part in determining the "transition" behaviour of the welded specimens. As yet the elucidation of this problem has not been approached experimentally, so that opinion at the present time is to a certain degree speculative. Three possible explanations have been advanced. One is based upon the fact that it is known that hydrogen in steel increases the transition temperature, and therefore with a rutile type of electrode there is the possibility of hydrogen diffusing into the parent plate and, at the base of the artificial notch, creating conditions more favourable to the initiation of failure. This may appear to be a little far-fetched, and the general opinion at the present time favours a more mechanical approach, based upon either micro-fissuring or macro-cracking in the weld metal bridging the two saw cuts in the plate material (one on either side of the weld). These two latter suggestions do, however, highlight the known fact that the role of the initiating defect is of major importance. As a consequence it is justifiable to ask what happens in a wide-plate test in the absence of an initiating defect. The answer is that brittle fracture at low applied loads does not occur. This is borne out by the lack of success encountered recently in the United States, where an attempt was made to develop a test specimen in the form of a small pressure vessel for the purpose of studying the brittle fracture problem. It did not prove possible to initiate brittle fracture in the pressure vessel until a wholly unacceptable design feature, the purpose of which was to provide a very major stress concentration, was incorporated in the vessel.

Such considerations, therefore, lead to the second inescapable pre-requisite for brittle fracture at low applied loads: there must be a defect in the weld zone, of sufficient magnitude in its influence, before fracture can be initiated.

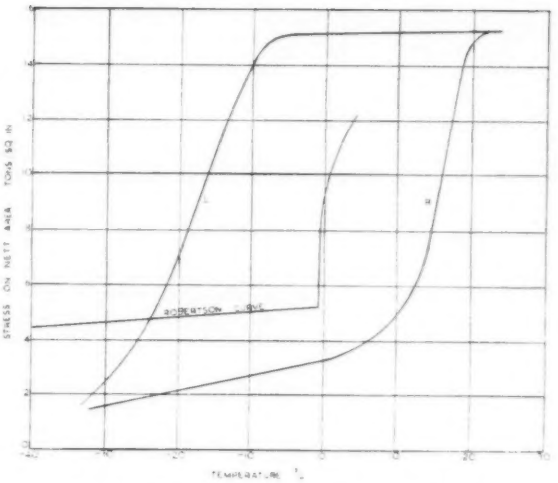


Fig. 4.—Wide plate test results for mild steel.

A statement of this type clearly summons before one's eyes a wide scope of experimental programmes to ascertain the quantitative aspects of the situation. But the question will necessarily arise as to whether the effort would be worth while. In pressure vessel construction, a potent tool in the form of radiography has been used by the manufacturers to ensure that gross defects do not exist in the pressure vessels when they are put into service. No one knows better than the experienced manufacturer the limitations of the non-destructive methods of examination he uses. It is well recognised within the industry that radiography is not necessarily competent to detect fine cracks in thick weldments. It is also recognised within the industry that fabrication methods play an important part in the incidence of cracks, and the reliable manufacturer will not use a cheaper method of construction if it is known to be prone to leave cracks or crack-like defects in the welded zone. With, therefore, the use of radiography and well-established and verified practices, the possibility of leaving in a butt weld a defect of a magnitude of sufficient severity to lead to failure at low applied stresses is extremely remote, so extreme in fact that one is inclined to say impossible. Enthusiasts for the use of ultrasonic inspection techniques are inclined to emphasise the fact that here is a tool that can dispel the fear of leaving any defect, no matter how small, in a weld no matter how thick. Whilst this may be true, the merits and demerits of alternative methods of non-destructive testing fall outside the scope of this address. It is proposed therefore to leave this subject with the final thought that, whatever may happen in the future, so far as can be ascertained there is no known case of brittle failure at low applied stress having occurred in a welded seam that had been radiographed.

All butt welds in Class I pressure vessels can be radiographically examined, otherwise the welds themselves could not be made. It would appear therefore that the possibility of such vessels failing by the brittle fracture mechanism is extremely remote, no matter what type of steel may be employed, because two of the necessary conditions for such failures, (*a*) residual stress, and (*b*) an initiating defect, are both absent: stress relief and non-destructive examination are demanded in all present-day

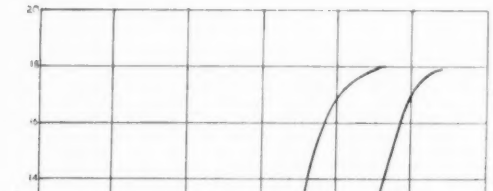


Fig. 5.—Wide plate test results for N.D.I. steel.

British specifications for such vessels. It is not surprising to find, therefore, that there is no known case of brittle fracture of a Class I pressure vessel in service, even though such vessels have been in service for many years at temperatures well below any conventional transition temperature of the materials involved.

In view of the agreement between extensive service experience of Class I pressure vessels and the laboratory investigations briefly described above, one is justified in asking what is the remaining problem in the experimental programme that has any importance to the manufacturer and user of pressure vessels, and why is so great an emphasis being placed upon steel selection by various competent bodies.

The manufacturer has two aspects to consider, the possibility of a brittle fracture, (*a*) during construction, and (*b*) for vessels that cannot be submitted to an effective stress-relieving operation. In both instances residual stress of a high magnitude will be present, and since there may exist the possibility of a defect of sufficient size to trigger off brittle fracture, it is necessary for the fabricator to consider carefully his choice of the steels available. In large constructions, where large tonnages of steel are involved, the economic aspects cannot be disregarded, for the extra price per ton of steel of improved notch ductility characteristics can be an appreciable percentage of the total manufacturing costs.

Fig. 5 is similar to Fig. 4 but is for a steel (N.D.I.) with somewhat better notch impact properties. Three effects are worthy of comment. The temperature difference between the two Wells curves is not so noticeable in Fig. 5 as in Fig. 3, suggesting that with an improvement in the steel the influence of the electrode type is not so prominent. Secondly, with the two sets of specimens with root runs made with the low-hydrogen electrodes there is no substantial difference between the two series of results. With the specimens welded with the rutile electrodes there is a shift towards lower temperatures for the steel with the better notch impact properties. The third point to notice is that although the Robertson transition temperature is not markedly changed, the major influence of the steel quality has been to raise the stress level for the crack-arrest temperatures below the transition temperature.

Arising from the changes in the relative positions of the wide-plate test results and the Robertson curve, it can be seen that with the improved steel a lower temperature and a higher stress is on the whole necessary to initiate catastrophic brittle fracture. This arises from the fact that, except for stresses below 6 tons/sq. in., the curves for the wide-plate tests are to the left and above the Robertson curve.

It is submitted, therefore, that we now have an adequate picture of the brittle fracture problem as it applies to pressure vessels, and it will perhaps be appreciated how futile it is to base the probability of failure on the conventional notch impact tests. This, of course, is not to imply that a notch-impact test may not have some bearing on the problem, but rather to emphasise that its importance is confined to a qualitative assessment. At the same time it is not suggested that we are in possession of all the desired facts. It is well recognised throughout the British industry that the next most important point to establish is the influence of section thickness. The truth of this statement lies in the ready response made, by those organisations approached, to a special appeal that was made on behalf of the British Welding Research Association for the necessary funds to extend their work to plates of 3 in. in thickness.

Also, further information is surely desirable on the significance of the type of defect producible in welded plates. It must be considered that the original defect explored by Wells is one that is not likely to simulate conditions that will exist in a well-constructed pressure vessel and it would be of considerable interest, and perhaps importance, to have a clearer understanding of the more precise mechanism by which the defect operates. Such tests are expensive and time-consuming and one has of necessity to curb one's impatience, but meantime it is submitted that the two researches on which our attention has been focused have advanced our understanding of the brittle fracture problem in pressure vessels to the point when definite use can be made of the data to the advantage of engineering industry.

To recapitulate the findings of the investigations mentioned, it can be seen that for brittle fracture at low applied loads to occur, three factors have to be present simultaneously, namely :

(*a*) there must be residual welding stresses ;

(*b*) there must be a defect present of sufficient severity that it can be the point of initiation of the fracture ; and

(*c*) the structure must be at a sufficiently low temperature, which cannot be defined by conventional methods of testing.

There is only one reservation that seems reasonable to make at this stage, and that is that we still do not know sufficient concerning the influence of stress concentrations of a serious magnitude. It would seem, therefore, that it is permissible to associate the above conclusions with conventional good practice in design.

Finally, some consideration might be paid to the importance of this work in the field of nuclear power production. Here two special considerations are thought

to apply. In this most recent of all power production methods, the containment vessel (the reactor vessel) of the radioactive core is, for the gas-cooled graphite moderated reactor, of a size and thickness that would appear at first sight to necessitate special treatment during manufacture. But is this, in fact, really such a problem? It is agreed that much of the construction has to be undertaken under site conditions, but it is also agreed that those site conditions can approximate quite closely to those appertaining in the average pressure-vessel factory. The difficulties are largely those imposed by the methods of mechanical handling and assembly. The difficulties, however, are not of an insuperable nature; adequate planning of the constructional methods, provided they are based on the well-recognised requirements of experienced pressure vessel manufacture, will be an adequate safeguard against the introduction of defects of sufficient magnitude and orientation to initiate brittle fracture. Furthermore, the steel qualities employed can be of a type that not only has adequate resistance to crack propagation, but is also characterised by freedom from laminar defects which, as is well known, can lead to the occurrence of more serious defects during welding.

For the containment vessels for other types of reactors, such as the pressurised water types, the problems are exactly those at present met with and overcome in present-day practice. The only major difference is that the thickness of the steel (possibly 6 to 8 in.) may exceed by a relatively small degree the thickness (5 to 6 in.) of vessels now quite frequently produced as a factory routine. In such instances the one factor that is of predominant importance is the adequacy of the non-destructive method of weld examination. This is a serious problem which does not need discussion at this stage, as it will suffice to state that adequate methods are now available in those shops where such work has been undertaken in the past.

The one remaining problem, therefore, in nuclear power production is the effect of irradiation by neutrons on the reactor vessel. The effect of neutron irradiation is becoming clearer as more experimental results are becoming available. Briefly, the evidence is that under neutron bombardment the transition temperature of a steel, measured by conventional notch impact tests, is increased. The increase is determined by the extent of the irradiation, both time and density, and by the speed of the neutrons. The greater the neutron velocity the more potent its influence. Also, the temperature of the steel has an important part to play. It would appear that the maximum "damage" to the steel can occur in the temperature range 150°-250° C., and that above 300° C. the damage can be annealed out.

Sufficient has probably been said, therefore, to indicate that the problem of the effects of neutron irradiation in reactors is not capable of simple solution until more accurate data can be obtained from actual service measurements of flux density. At the moment estimates can be made, and it has been stated that an increase of the transition temperature—determined from Charpy V-notch impact specimens—can be expected to be as small as a few degrees Centigrade or as large as 40° C. in the case of the reactor vessels of the gas-cooled graphite-moderated reactors at present under construction in this country.

On the basis of the Wells wide-plate tests so sketchily described above, we find from Figs. 3 and 5 that for the welds incorporating root runs made with low-hydrogen electrodes that the transition temperature has barely changed, whereas the change in the transition temperature for the steel, determined by Charpy tests, was of the order of 25° C. Or again, if comparisons are drawn between the behaviours of the ordinary mild steel of Fig. 3 and a notch ductile steel of the N.D.IV type, where by conventional Charpy tests the transition temperature is decreased by approximately 80° C., it is found in the Wells tests that the transition curve has been shifted only 15–20° C.

If the estimates of irradiation damage are reasonably accurate, and if the corresponding data determined as the result of the welded wide-plate tests can be relied upon, it is clear that with present-day good practice in design, workmanship, construction and erection, and with steels that have a long experience of industrial usage, there is no need to anticipate brittle failures in nuclear reactor vessels now being produced for industrial power production.

Birlec in South Africa

CONSOLIDATING their venture into the South African market made early in 1956, Birlec, Ltd., announce that a Johannesburg branch office, under the management of Mr. S. G. King, has now been formed. Situated between Boksburg and Benoni, the new branch will deal with all enquiries, including project design and engineering service work. The postal address is P.O. Box 99 Witfield, Transvaal.

It is expected that the chief demand will be for melting furnaces, particularly for those used mainly in making alloy steels. Whilst certain special equipment and parts will continue to be imported from the main Birmingham works, the majority of the equipment will be manufactured locally by the Firlst Electro Corporation. Several major orders have been placed during the last two years.

In the Federation of Rhodesia and Nyasaland the British Thomson-Houston Co. (Central Africa) (Pvt.), Ltd., will continue to act as Birlec agents, but the services of the new branch will now be fully available to Birlec furnace users in the Federation, particularly for technical advice and assistance when required.

Bigger E.R.W. Tubes

A NEW electric resistance weld tube mill, which can produce E.R.W. steel tubes up to 5½ in. in diameter, with a wall thickness of ¼ in. and in lengths up to 60 ft., has been designed by the Tube Investments subsidiary, Tube Products, Ltd., and is now in operation at the company's Falcon Works at Wednesfield.

Previously, E.R.W. tubes made in this country have not exceeded 4½ in. in outside diameter. The new mill holds promise of important applications for E.R.W. tubes, which can now be made in the increased size range for boiler tubes to B.S. 1654, ferrous tube to B.S.806 Class H, mechanical tubes to B.S. 1775 and tubes for the motor car industry to B.S. 980. The mill can produce the new range of big tubes with a high standard of concentricity and surface finish.

ACHESON INDUSTRIES (EUROPE), LTD., have removed from 18 Pall Mall to 1 Finsbury Square, London, E.C.2. (telephone: MONarch 5811 (4 lines); Telegraphic Address: Oildag, Stock, London; Cable Address: Oildag, London).

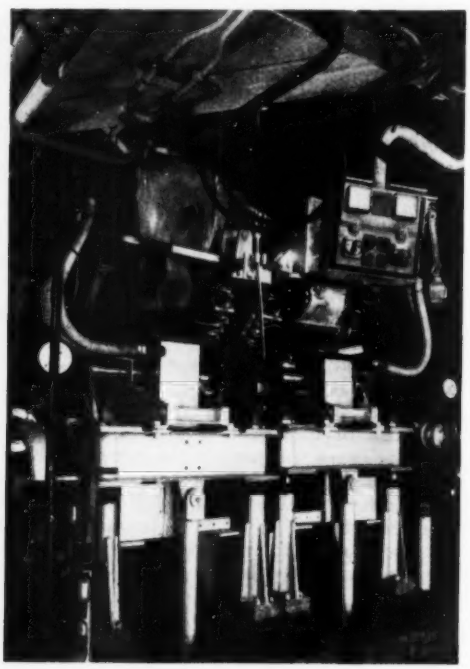
Twin-Strand Continuous Steel Casting at Barrow

THE continuous casting machine at Barrow Steel Works, Ltd., where experiments in the continuous casting of steel have been in progress since 1952, has been modified so that twin-strand continuous casting of billets and small slabs is now possible. The modified machine, which has double the capacity of the former unit and is equipped with automatic control, operates at speeds of between 220 and 360 in./min. when casting 2 in. square billets at least twice as fast as any other known continuous casting plant. Normal performance with billets of this size is a production rate of 15 tons/hr. Despite these high casting speeds, the machine is only about 29 ft. tall to the top of the mould, the latter being 32 in. long.

Hot metal for the process is supplied from a 7½-ton capacity electric arc furnace, which is tapped into a preheated ladle of the same capacity. Steel is teemed from the ladle into two separate tundishes simultaneously, a small transverse launder being used to divide the metal supply. The tundishes, like the ladle, are preheated and fired during casting by means of specially-developed oil burners. An anti-swirl nozzle of Barrow design fitted to the bottom of each tundish ensures an even and steady flow of metal into the mould.

On emerging from the reciprocating copper mould, the billet passes through a series of water sprays and is solidified before it reaches the withdrawal rolls. At the base of either strand there is a single pair of rolls actuated by a power cylinder which bends the billet in order to discharge it horizontally on to a roller path, where it is straightened under its own weight and then cut to length. The extent of the cooling bank facilities available determines the length of the billet; lengths of up to 150 ft. have been obtained, though 30 ft. billets or shorter are standard practice.

General view of the twin-strand continuous casting plant, showing billets being cut to length for transfer to the cooling banks.



This picture of the continuous casting plant shows the arrangement of the two tundishes and the copper moulds.

Automatic control devices are employed to stabilise the level of the liquid metal in the mould during casting. This leads to greater reliability in operation as the entire production sequence proceeds automatically from the moment when casting begins; it also aids uniformity and consistency in the finished product. By giving greater flexibility, the twin-stranding arrangements ensure greater reliability and little or no loss of yield. Because of the close temperature control exercised, casting times may last up to 1¼ hours; longer casting periods are feasible and for a 7½-ton cast have been extended to two hours without adverse effects on the steel.

The twin-strand plant at Barrow is designed to produce billets from 2 in. to 4 in. square and slabs up to 12 in. × 4 in. Much of the experience gained so far has been in the range of carbon steels containing between 0·05 and 0·85% carbon. For these steels, the average yield of commercially sound material runs between 98 and 99%. Equally good yields have been obtained with low alloy steels, including silico-manganese spring steel, high sulphur free-cutting steel, and stainless steels. In subsequent rolling and forging processes, continuously cast billets and slabs have shown physical properties fully comparable with those produced by conventional methods.

Properties of a Large Extrusion Supplied to Specification D.T.D. 363

By T. R. G. William, M.Sc., A.I.M.,* S. W. Hallwood† and R. W. Hayden‡

The authors present the results of a programme of static and fatigue testing on a large heat treated extrusion in an Al-Zn-Mg-Cu type alloy supplied to D.T.D. 363. Longitudinal test pieces taken from the centre of the extruded section showed a lower tensile strength than those located nearer the surface. It is suggested that the most effective method of reducing this variation would be by overcoming the mass effect in quenching.

NEARLY all high strength aluminium alloys exhibit the " extrusion " or " press " effect which gives them higher longitudinal proof and tensile strengths than the corresponding sheet, wire and forged products. Zeeman[1] defines the press effect as the excess raising of the longitudinal strength of extruded material with texture, as compared with recrystallised material, and states that this enhanced longitudinal strength along the extrusion fibre is lost during recrystallisation. Several explanations of the high longitudinal strength of extrusion fibre have been proposed and Hardy[2], in a review of these theories, considers Unckel's claim that preferred orientation is the cause of the high properties, to be the most satisfactory explanation.

Most specifications for extruded products have been based upon mechanical properties obtained on test pieces containing at least some of this extrusion texture. In thick sections the recrystallised zone of coarse grain forms a distinct outer band (see Fig. 1), and by limiting the depth of this band to about $\frac{1}{8}$ in., mechanical test pieces prepared from the outside position of the extrusion can be completely free of this lower strength material. With thin sections (less than $\frac{3}{8}$ in.), however, it is impossible to avoid some of the recrystallised material (see Fig. 2), and special provision has to be made in the specifications to allow a lower property requirement limit for such a material, as this excerpt from specification D.T.D. 363 shows :—

	0·1% Proof Stress (tons/sq. in.)	Ultimate Tensile Stress (tons/sq. in.)	Elongation (%)
Minimum strength requirement for material less than $\frac{3}{8}$ in. thick	30	35	7
Minimum strength requirement for material $\frac{3}{8}$ in. thick and above	33	38	5

As the size of the extrusion presses and of the sections

* Lecturer in Metallurgy, Southampton University.
† Metallurgist, Folland Aircraft, Ltd.
‡ Assistant Metallurgist, Folland Aircraft, Ltd.

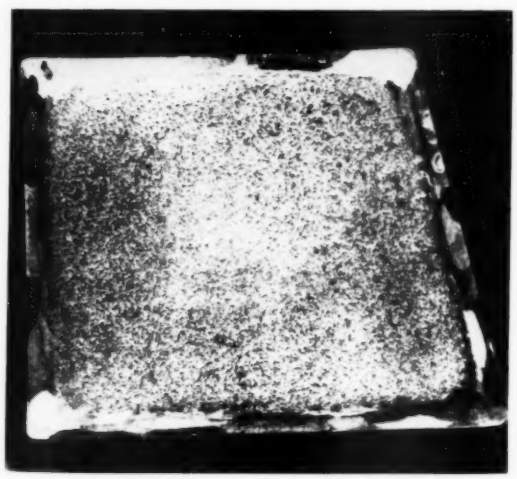


Fig. 1. Macrostructure of transverse slice through an extrusion. Etching in caustic soda has revealed the fibrous grain structure surrounded by a coarse grain outer band. × 1

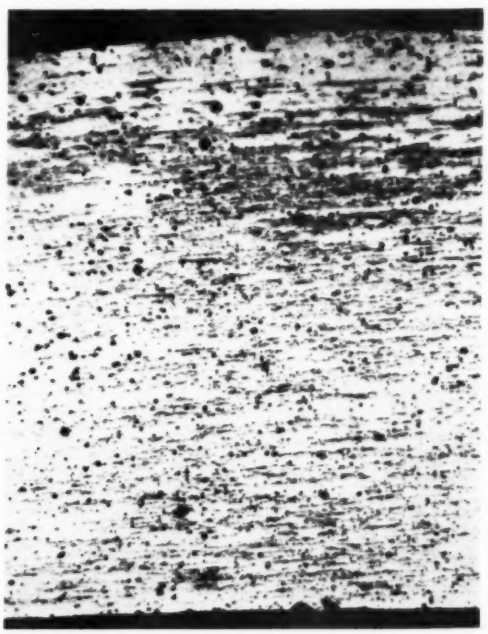


Fig. 2. A photomicrograph showing a mixture of fibrous and recrystallised grain in a 'thin' section extrusion, revealed by etching in 25% nitric acid at 70° C. for 2 minutes. There is no distinct line of demarcation between the two different grain structures in this sample. × 100

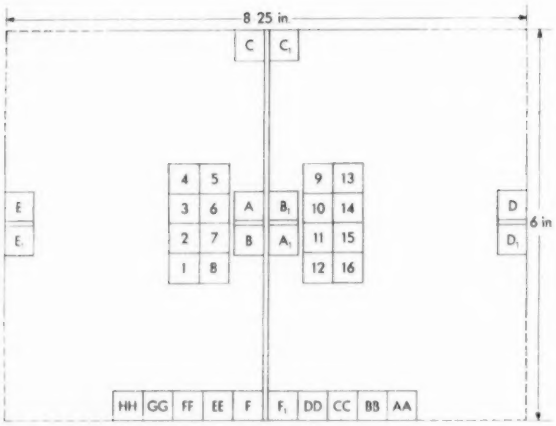


Fig. 3.—Location of longitudinal tensile test pieces shown on a cross section of block 1.

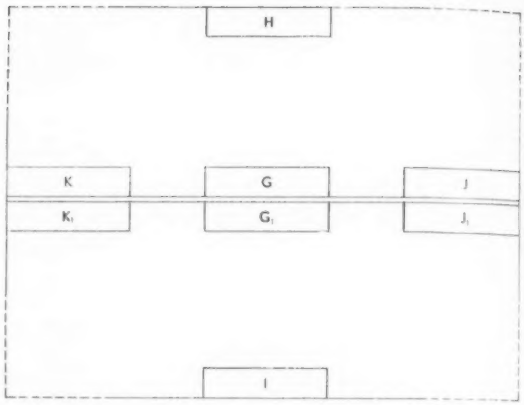


Fig. 4.—Location of long transverse tensile test pieces shown on a cross section of block 2.

produced have increased, it has been found that test pieces located near the outside surface of the extrusions have been giving results considerably higher than those of tests carried out at the centre, and provision is now made in specifications for special tests to be performed to assess the variation encountered (see L.100 Section 6).

This programme of testing was undertaken to assess the static and fatigue strength of a large extrusion section in the Al-Zn-Mg-Cu type alloy, supplied to specification D.T.D. 363 requirements. The extrusion was obtained in the fully heat treated and aged condition, and Figs. 3–6 show the circumscribing rectangle of the section, the total area of the solid extrusion being 42 sq. in.

Experimental Work

The static strength was ascertained with a 5½ ton Denison machine, using the Houndsfield extensometer and test pieces 0·252 in. diameter. The samples were prepared from the extrusion in three directions at right angles—longitudinally in the direction of extrusion, and parallel to the major and minor axes of a transverse section through the extrusion. The location of the test pieces can be seen in Figs. 3–7. Table I provides the results obtained in these tests, and Table II the variation in the properties of the extrusion at different locations.

A further batch of samples was cut from the extrusion, adjacent to the first batch, and these were marked with the suffix 1, as shown in Figs. 3–6. The samples were then re-solution heat treated in test piece form at 465° C. for 1 hour, quenched in cold water, and aged for 18 hours at 120° C. Table III shows the results obtained and Table IV the alteration in properties.

Chemical analysis of samples drilled from the " centre " and the " outside " layers of the extrusion gave the following results :—

	Cu%	Mg%	Zn%	Cr%	Mn%	Ti%	Fe%	Si%
Outside	1·99	2·37	7·39	0·15	0·15	0·06	0·28	0·17
Centre	1·98	2·10	6·9	0·13	0·14	0·075	0·25	0·20

Rolls Royce machine test pieces were prepared to assess fatigue properties at the " centre " and " outside " positions in the longitudinal direction (Fig. 3), and in the " centre " position in the short transverse direction (Fig. 5). The results of these fatigue tests are shown in Table V, and have been plotted graphically in Fig. 8.

One inch square samples were cut from the " centre " and " edge " of the extrusion and these were forged at 460° C. along the fibre direction to ½ in. square test pieces. They were then solution heat treated at 465° C. for 1 hour, aged for 18 hours at 120° C., and their static strength determined as well as the metallographic

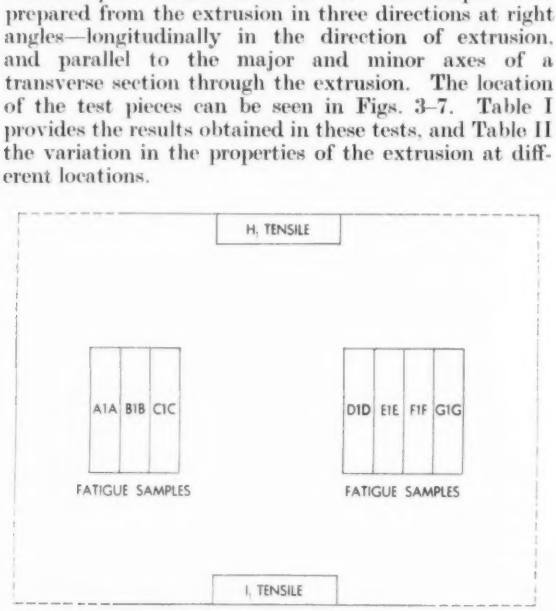


Fig. 5.—Location of short transverse fatigue test pieces shown on a cross section of block 3.

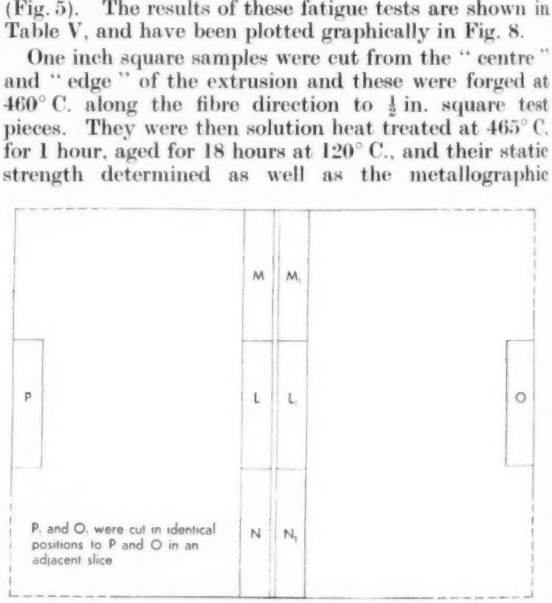


Fig. 6.—Location of short transverse tensile test pieces shown on cross sections of blocks 4 and 5.

TABLE I.—PROPERTIES OF EXTRUDED SPAR IN Al-Zn-Mg-Cu ALLOY.

Test Piece		0·1% Proof Stress (tons/sq. in.)		Ultimate Tensile Stress (tons/sq. in.)		Elongation (% on $4\sqrt{A}$)	
Location	Identity						
		Longitudinal Direction					
Outside	C	35·6	average	42·3	average	13·3	average
	D	38·7		42·8		11·8	
	E	38·4	37·2	42·5	42·4	12·0	12·1
	F	36·2		41·8		11·3	
Centre	A	27·2	average	34·0	average	8·3	average
	B	27·9	27·6	35·0	34·5	11·5	9·9
		Long Transverse Direction					
Outside	H	32·8	average	38·8	average	6·6	average
	I	32·9	32·9	38·8	38·8	7·0	6·8
Intermediate	K	29·0	average	32·0	average	1·4	average
	J	28·5	28·8	33·0	32·5	1·8	1·6
Centre	G	23·6		28·8		4·6	
		Short Transverse Direction					
Outside	P	29·8	average	38·4	average	4·9	average
	O	29·8	29·8	38·6	38·5	5·3	5·1
Intermediate	M	23·9	average	28·6	average	1·8	average
	N	22·7	23·3	28·0	28·3	1·5	1·7
Centre	L	21·8		26·7		1·9	

TABLE II.—VARIATION IN PROPERTIES THROUGHOUT THE SPAR.

Details of Tests	Item	0·1% Proof Stress (tons/sq. in.)	Ultimate Tensile Stress (tons/sq. in.)	Elongation (% on $4\sqrt{A}$)
Longitudinal Direction at Outside Position		37·2	42·4	12·1
Longitudinal Direction at Centre Position		27·6	34·5	9·9
Variation	(*a*)	9·6	7·9	2·2
Long Transverse Direction at Outside Position		32·9	38·8	6·8
Long Transverse Direction at Centre Position		23·6	28·8	4·6
Variation	(*b*)	9·3	10·0	2·2
Short Transverse Direction at Outside Position		29·8	38·5	5·1
Short Transverse Direction at Centre Position		23·3	28·5	1·7
Variation	(*c*)	6·5	10·0	3·4
Longitudinal Direction at Outside Position		37·2	42·4	12·1
Long Transverse Direction at Outside Position		32·9	38·8	6·8
Variation	(*d*)	4·3	3·6	5·3
Longitudinal Direction at Centre Position		27·6	34·5	9·9
Long Transverse Direction at Centre Position		23·6	28·8	4·6
Variation	(*e*)	4·0	5·7	5·3
Longitudinal Direction at Outside Position		37·2	42·4	12·1
Short Transverse Direction at Outside Position		29·8	38·5	5·1
Variation	(*f*)	7·4	3·9	7·0
Longitudinal Direction at Centre Position		27·6	34·5	9·9
Short Transverse Direction at Centre Position		23·3	28·3	1·7
Variation	(*g*)	4·3	6·2	8·2
Long Transverse Direction at Outside Position		32·9	38·8	6·8
Short Transverse Direction at Outside Position		29·8	38·5	5·1
Variation	(*h*)	3·1	0·3	1·7
Long Transverse Direction at Centre Position		23·6	28·8	4·6
Short Transverse Direction at Centre Position		23·3	28·3	1·7
Variation	(*i*)	0·3	0·5	2·9

structure. Table VI shows the static test results obtained, and the metallographic structure is shown in Fig. 9.

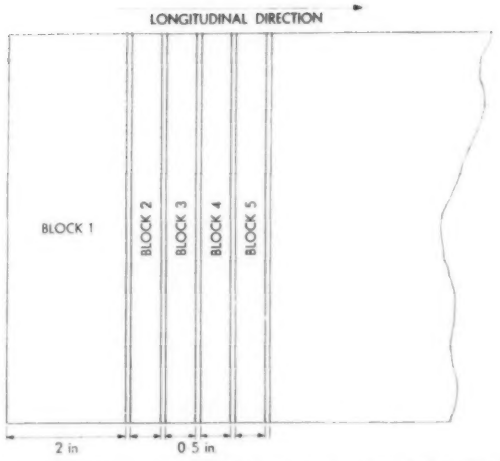


Fig. 7.—Location of blocks 1–5 relative to the length of the extrusion.

TABLE III.—EFFECT OF RE-SOLUTION HEAT TREATMENT.*

Test Piece		0·1% Proof Stress (tons/sq. in.)		Ultimate Tensile Stress (tons/sq. in.)		Elongation (% on $4\sqrt{A}$)	
Location	Identity						
		Longitudinal Direction					
Outside	C1	36·3	average	42·6	average	13·3	average
	D1	37·6		43·3		11·8	
	E1	37·0	37·2	43·0	42·9	12·0	12·1
	F1	35·7		42·6		11·3	
Centre	A1	34·8	average	40·2	average	11·9	average
	B1	34·4	34·6	40·2	40·2	11·5	11·7
		Long Transverse Direction					
Outside	H1	31·5	average	38·8	average	9·5	average
	I1	30·5	31·0	38·9	38·9	9·2	9·4
Intermediate	K1	28·6	average	33·2	average	1·3	average
	J1	28·8	28·7	34·0	33·6	2·0	1·7
Centre	G1	28·0		33·5		2·2	
		Short Transverse Direction					
Outside	P1	31·5	average	38·2	average	7·3	average
	O1	31·4	31·5	38·6	38·4	8·0	7·7
Intermediate	M1	29·0	average	32·4	average	1·4	average
	N1	29·6	29·3	32·7	32·6	1·0	1·2
Centre	L1	27·7		30·2		1·6	

* ½-in. square and 2 in. long test pieces were cut from various positions in the spar and re-solution heat treated at 465° C. for 1 hour, quenched in water, and aged for 18 hours at 120° C.

Results

The macro-structure of a part of a transverse section through the extrusion is shown in Fig. 10. The extrusion is almost completely free of a recrystallised outer band of coarse grain, and the structure is typical of the

TABLE IV.—ALTERATION IN PROPERTIES RESULTING FROM RE-SOLUTION HEAT TREATMENT.

Details of Tests	Item	Before Re-solution Heat Treatment: 0·1% Proof Stress (tons/sq. in.)	Before Re-solution Heat Treatment: Ultimate Tensile Stress (tons/sq. in.)	Before Re-solution Heat Treatment: Elongation (% on $4\sqrt{A}$)	After Re-solution Heat Treatment: 0·1% Proof Stress (tons/sq. in.)	After Re-solution Heat Treatment: Ultimate Tensile Stress (tons/sq. in.)	After Re-solution Heat Treatment: Elongation (% on $4\sqrt{A}$)
Longitudinal Direction at Outside Position	(*j*)	37·2	42·4	12·1	37·2	42·9	12·1
Variation due to Treatment					0	+0·5	0
Longitudinal Direction at Centre Position	(*k*)	27·6	34·5	9·9	34·6	40·2	11·7
Variation due to Treatment					+7·0	+5·7	+1·8
Long Transverse Direction at Outside Position	(*l*)	32·9	38·8	6·8	31·0	38·9	9·4
Variation due to Treatment					−1·9	+0·1	+2·6
Long Transverse Direction at Centre Position	(*m*)	23·6	28·8	4·6	28·0	33·3	2·2
Variation due to Treatment					+4·4	+4·5	−2·4
Short Transverse Direction at Outside Position	(*n*)	29·8	38·5	5·1	31·5	38·4	7·7
Variation due to Treatment					+1·7	−0·1	+2·6
Short Transverse Direction at Centre Position	(*o*)	21·8	26·7	1·9	27·7	30·2	1·6
Variation due to Treatment					+5·9	+3·5	−0·3
Longitudinal Direction at Outside Position		37·2	42·4	12·1	37·2	42·9	12·1
Longitudinal Direction at Centre Position		27·6	34·5	9·9	34·6	40·2	11·7
Variation due to Position	(*p*)	9·6	7·9	2·2	2·6	2·7	0·4
Long Transverse Direction at Outside Position		32·9	38·8	6·8	31·0	38·9	9·4
Long Transverse Direction at Centre Position		23·6	28·8	4·6	28·0	33·3	2·2
Variation due to Position	(*q*)	9·3	10·0	2·2	3·0	5·6	7·2
Short Transverse Direction at Outside Position		29·8	38·5	5·1	31·5	38·4	7·7
Short Transverse Direction at Centre Position		21·8	26·7	1·9	27·7	30·2	1·6
Variation due to Position	(*r*)	8·0	11·8	3·2	3·8	8·2	6·1

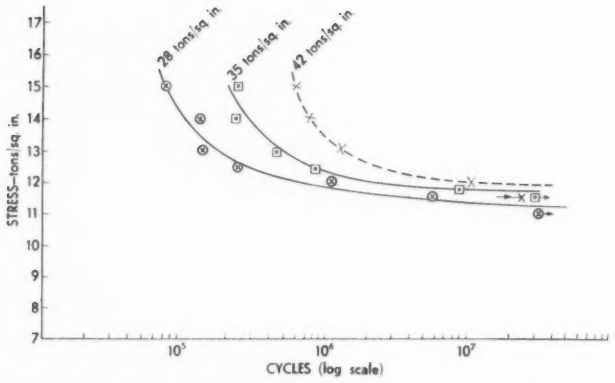


Fig. 8.—Results of fatigue tests in the short transverse direction.

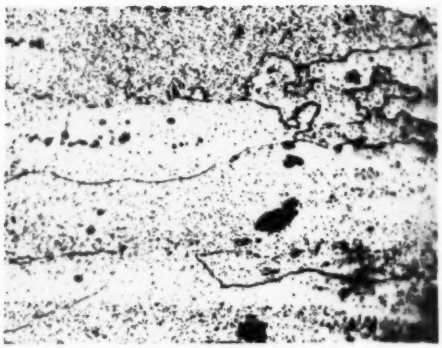


Fig. 9.—Recrystallised grain structure of forged and heat treated material, revealed by etching in Wasserman's reagent. × 100

fibrous grain exhibiting the "press" or "extrusion" effect. The absence of an outer band in the Al-Zn-Mg-Cu alloys is not unusual, and Smith[3] states that the action of chromium and manganese in these alloys in preventing recrystallisation is facilitated by the fact that the solution treatment temperature is about 50° C. lower than that of most other alloys.

TABLE V.—RESULTS OF FATIGUE TESTS.

Details of Tests	Identity	Diameter (in.)	Stress (tons/sq. in.)	Load (lbs.)	Cycles	Comments
Longitudinal Direction at Outside Position	CC	0·161	14	1·51	747,300	Fractured
	BB	0·161	13	1·40	1,295,100	"
	EE	0·1605	12	1·28	10,298,800	"
	FF	0·1605	11	1·17	42,733,400	Unbroken
	GG	0·1605	11·5	1·22	24,466,100	"
	AA	0·1605	15·0	1·60	627,200	Fractured
	HH	0·1605	12·5	1·33	5,991,500	"
Longitudinal Direction Near Centre	12	0·1605	15	1·6	234,400	Fractured
	16	0·161	14	1·51	235,300	"
	11	0·161	13	1·4	451,300	"
	14	0·160	12	1·26	9,140,300	"
	3	0·1605	11	1·17	28,416,500	Not broken
	6	0·1605	11·5	1·22	26,014,200	"
	5	0·1605	12·5	1·33	881,300	"
Long Transverse Direction Near Centre	G1G	0·1605	15·0	1·6	72,600	Fractured
	F3F	0·161	14·0	1·51	128,200	"
	A1A	0·1605	13·0	1·38	138,100	"
	B1B	0·1605	12·5	1·33	235,100	"
	D1D	0·1610	12·0	1·3	1,086,600	"
	C1C	0·1605	11·5	1·22	1,561,700	"
	E1E	0·1605	11·0	1·17	23,106,400	Not fractured

From Table I it can be seen that the highest static properties were obtained in the longitudinal direction and at the "outside" position, whilst the lowest properties were obtained in the short transverse direction at the centre of the extrusion, giving a maximum variation in properties throughout the extrusion of :—

0·1% Proof Stress (tons/sq. in.)	Ultimate Tensile Stress (tons/sq. in.)	Elongation (% on $4\sqrt{A}$)
13·9	14·1	10·4

Item (*d*) in Table II shows that the strength across the

TABLE VI.—EFFECT OF FORGING ON PROPERTIES OF THE ALLOY.

Details of Tests	0·1% Proof Stress (tons/sq. in.)	Ultimate Tensile Stress (tons/sq. in.)	Elongation (% on $4\sqrt{A}$)
As extruded LO and after re-solution heat treatment and ageing	37·2	42·9	12·1
As forged LO and after solution heat treatment and ageing	32·0	38·9	12·5
Difference in properties due to recrystallisation	5·2	4·0	0·4
As forged LC and after re-solution heat treatment and ageing	34·6	40·2	11·7
As forged LC and after solution heat treatment and ageing	30·6	37·0	11·9
Difference in properties due to recrystallisation. ..	4·0	3·2	0·2

LO = at outside position, longitudinal direction.
LC = at centre position, longitudinal direction.

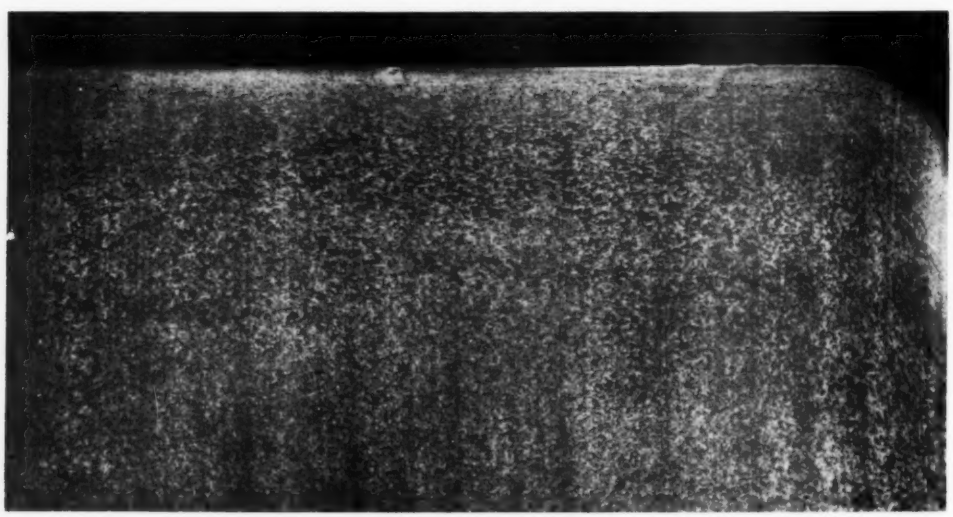


Fig. 10.—Macrostructure of a transverse slice through the extruded spar revealed by etching in 0·5% HF. ×1½

grain fibre at the outside position in the extrusion is lower than the strength along the grain fibre by :—

0·1% Proof Stress (tons/sq. in.)	Ultimate Tensile Stress (tons/sq. in.)	Elongation (% on $4\sqrt{A}$)
4·3	3·6	5·3

The cooling conditions during quenching at this outside position are uniform, and so also are the chemical composition and the size and distribution of the intermetallic compounds. The variation in properties noted at this outside position can therefore be attributed to the effect of grain orientation.

Variations in properties between centre and outside test pieces are large in all three directions tested (see Table II). When test pieces cut from the extrusion are re-solution heat treated as small test pieces to overcome the non-isothermal cooling obtaining in the extrusion quenched *en masse* the centre test pieces give considerably higher properties (Table IV). The difference between outside and centre test pieces is reduced by re-solution heat treatment and ageing as follows :—

	0·1% Proof Stress (tons/sq. in.)	Ultimate Tensile Stress (tons/sq. in.)	Elongation (% on $4\sqrt{A}$)
Difference before solution heat treatment, tested in longitudinal direction	9·6	7·9	2·2
Difference after solution heat treatment, tested in longitudinal direction	2·6	2·7	0·4
Difference due to mass effect	7·0	5·2	1·8

Thus, non-isothermal cooling during full scale production heat treatment is the major factor responsible for the variation in properties between outside and centre pieces.

Chemical analysis shows the outside position of the extrusion to be richer in zinc and magnesium than the centre. This variation probably arises from the inverse segregation effects in the cast billet. The flow pattern in the extrusion process transfers the outer layers of the billet so that they become the outer zones of the extrusion, and therefore tends to preserve any chemical heterogeneity present on a macro-scale in the cast billet (see Figs. 11 and 12).

A further way in which the flow pattern in extrusion may contribute to variations in the properties of the

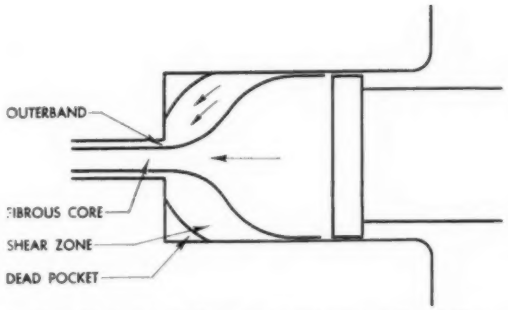


Fig. 11.—Diagram of flow pattern in extruding billet.

extrusion is that intermetallic constituents in the outer-layers of the billet are heavily fragmented during flow through the shear zones of the extruding billet, and are therefore finely dispersed and readily absorbed during solution heat treatment, whereas the intermetallics at the centre of the billet are only slightly deformed during extrusion and remain as coarse segregates in the extrusion. Absorption of these intermetallics by the matrix during heat treatment is slow. Fig. 13 shows such a coarse segregation of intermetallics near the centre of the extrusion.

Samples cut from the " centre " and " outside " of the extrusion and forged in the direction of the extrusion fibre showed the extrusion or press effect (Table VI) to be :—

	0·1% Proof Stress (tons/sq in.)	Ultimate Tensile Stress (tons/sq. in.)	Elongation (% on 4/A)
Edge	5·2	4·0	2·1
Centre	4·0	3·0	0·6

Fig. 8 shows the results of the fatigue tests. All the S/N fatigue curves flatten out at low stress levels and seem to exhibit a fatigue limit. At low cycles, the material of high tensile strength possesses a superior fatigue endurance to material of low tensile strength. The endurance of all three different samples are similar at 10^7 cycles and above, however.

Figs. 14 and 15 show the path of the fatigue crack through the main-structure of sample No. 14 prepared from material near the centre of the extrusion. In Fig. 14

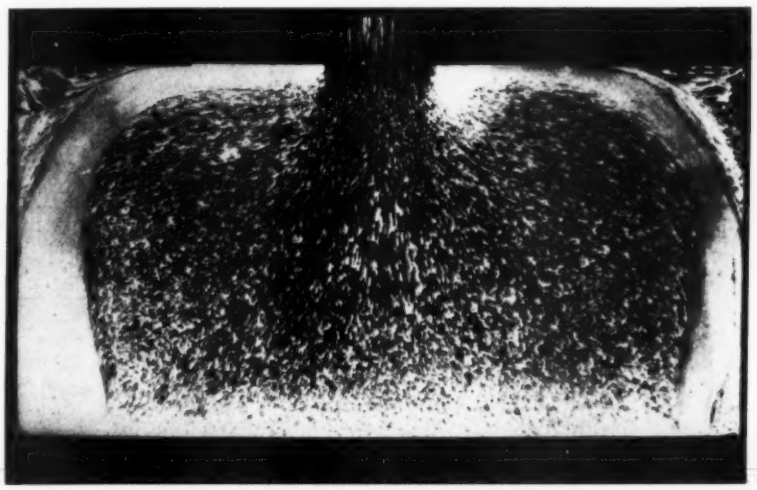


Fig. 12.—Macrostructure of an extruding billet in the direct extrusion process. Cast material moving into the die aperture through the shear zones is severely wrought and fragmented, whereas material at the centre of the billet is only elongated. Etched in NaOH. × ⅔

Fig. 13.—Coarse intermetallic segregate at centre of spar. × 1208

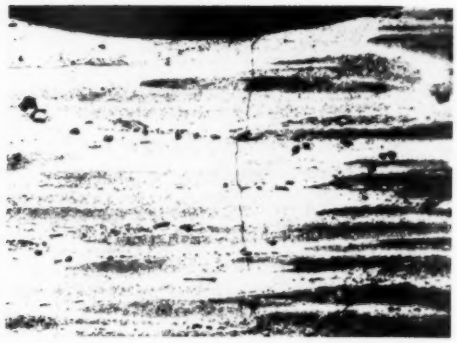


Fig. 14.—Microstructure showing path of fatigue crack through the elongated grain structure of the extrusion. Etched in Wasserman's reagent. × 100

the elongated grains are seen and the fatigue crack runs across these grains. At high magnification—Fig. 15—a sub-grain structure is revealed in the white zones denuded of finely dispersed intermetallics, and the fatigue cracks are transcrystalline to these sub-grains.

These results may be summarised as follows :—

(1) The highest static strength is obtained in the direction of the extrusion fibre. Across the fibre the proof and tensile strengths are similar to those of re-crystallised material except that the elongation values are lower.

(2) There is a large variation in the static strength properties of this material, the lowest properties in all the three principal directions being at the centre.

(3) Non-isothermal cooling conditions throughout the extrusion during quenching from solution heat treatment temperature constitute the major factor responsible for this variation in properties between " outside " and " centre " positions.

(4) The material of high tensile strength gives superior fatigue endurance at low cycles, but at 10^7 cycles and above the fatigue behaviour of fibrous material in the tensile strength range 28–42 tons/sq. in. is similar, all the curves tending to flatten out and to exhibit a fatigue limit.

General Conclusion

Extrusions are usually stressed in the longitudinal direction along the extrusion fibre and, despite the benefits of the extrusion effect, the loss of tensile strength in the centre of large sections is a serious limitation, particularly when the higher strength outer layers are machined away in fabrication. The most effective method of reducing this variation would be to overcome the " mass effect " in quenching. Two possible ways of accomplishing this are now being investigated :—

(*a*) increasing the cooling rate in water quenching by dissipating the steam envelope : and

(*b*) altering the composition of the alloy to render the effect of cooling rate in quenching on the static strength properties less critical.

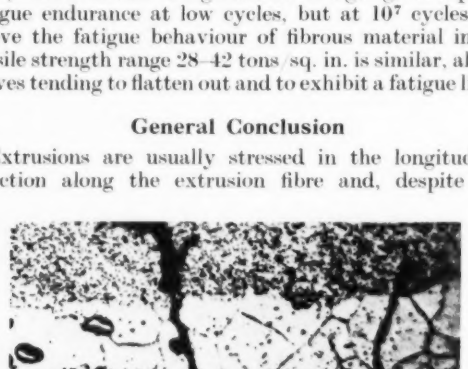


Fig. 15.—Microstructure at high magnification revealing the transgranular path of the fatigue cracks through the sub-grain structure of the white ' denuded ' zone in the extruded grain structure. Etched in Wasserman's reagent × 750

Acknowledgments

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REFERENCES

1 Zeeman, H. M., *Z.V.D.I.*, 1949, **91**, 621–626.
2 Hardy, H. K., *Metallurgia*, 1944, **30**, 240–244.
3 Smith, C. S., *J.I.M.*, 1950, **76**, 429–451.

Carblox, Ltd., Acquired

THOMAS MARSHALL & CO. (LOXLEY), LTD., of Storrs Bridge Works, Loxley, Near Sheffield has acquired the half interest previously held by The Morgan Crucible Co., Ltd., in Carblox Ltd., which now becomes a wholly owned subsidiary of Marshall. The members of Carblox Board will be Messrs. T. Marshall, A. Marshall and W. H. Crawshaw. Mr. D. W. Hubbard will continue as Sales Manager and Mr. P. S. Howgate, will continue as Works Manager. The main effect of the change will be that the Sales Department will now operate from the works at Loxley, which will ensure prompt attention to all customer requirements including full technical services.

Thomas Marshall & Co. (Loxley), Ltd., first supplied fireclay and fireclay refractories to the steel industry more than 100 years ago. In 1928 hot face insulation was developed and the subsidiary company, Kingscliffe Insulating Products, Ltd., was formed in 1936. In 1941 the carbon refractories used primarily for lining blast furnaces were produced, and Carblox, Ltd., was formed in 1945. Other subsidiaries are Ffrith Fireclay Co., Ltd., and The Hoyland Brick Co. (Marshall), Ltd.

Magnesia from Sea Water

Important Source of Raw Material for Refractories

Sand trap and sea water storage tanks.

THIS year marks the coming-of-age of the extraction of magnesia from sea water in this country, for it was in 1937 that The Steetley Company, Limited, installed a pilot plant to produce refractory magnesia by reacting burnt dolomite with treated sea water. The Steetley Company's interest in dolomite—a double carbonate of lime and magnesia—goes back as far as the 1880's, when it was first quarried by the Company at Steetley in Nottinghamshire. After dead-burning, it was supplied to steelmakers to line the furnaces for the newly-developed basic steelmaking process, which now accounts for 90% of the steel produced in Britain today.

In 1936, the Steetley Company pioneered a major technical development when, after intensive research, it was shown that a controlled addition of serpentine produced a burnt dolomite resistant to attack by atmospheric moisture, thus extending its use as a refractory material in the form of stabilised dolomite clinker, bricks and cements.

Today, the Steetley Company is believed to be the largest producer of refractories in Europe and the Commonwealth, with an annual production of 800,000 tons of finished refractories and 400,000 tons of other materials. Of the three divisions operating in the United Kingdom, the Dolomite Division is concerned with the extraction and treatment of dolomite for use in basic refractories and in rammed furnace hearths—some of the finer material is used in coal mining and agriculture; the Magnesia Division is occupied entirely with the production of Britmag magnesia from sea water; and the Refractories Division produces a wide range of brick products—stabilised dolomite, magnesite, chrome-magnesite, magnesite-chrome, silica and sillimanite—for almost all industries using high temperature processes. There is also a Canadian Division, operating a dolomite plant at Hamilton, Ontario, and crushed and cut stone plants in South-Western Ontario.

Importance of Sea Water Magnesia

Current production capacity of magnesia from sea water at the Hartlepool plant of the Steetley Company is in the region of 150,000 tons a year, but in 1937, although the process had been proved to be technically possible by bathtub experiments, Steetley's Chairman and Managing Director, Mr. N. M. Peech, encountered a good deal of scepticism from those who considered it was unnecessary to go to such lengths to obtain a material which could be won by simple mining operations in Austria.

Britain has no indigenous supplies of magnesite, and the strategic importance of the new plant was soon realised when war became imminent. A full-scale plant—the world's first sea water refractory magnesia plant—was laid down in 1938, which was producing at the annual rate of 4,000 tons by 1939. Development thereafter was rapid, as large quantities of magnesia were required not only for refractories, but also for the production of magnesium metal for incendiary bombs and the various magnesium alloys used in the aircraft and other industries.

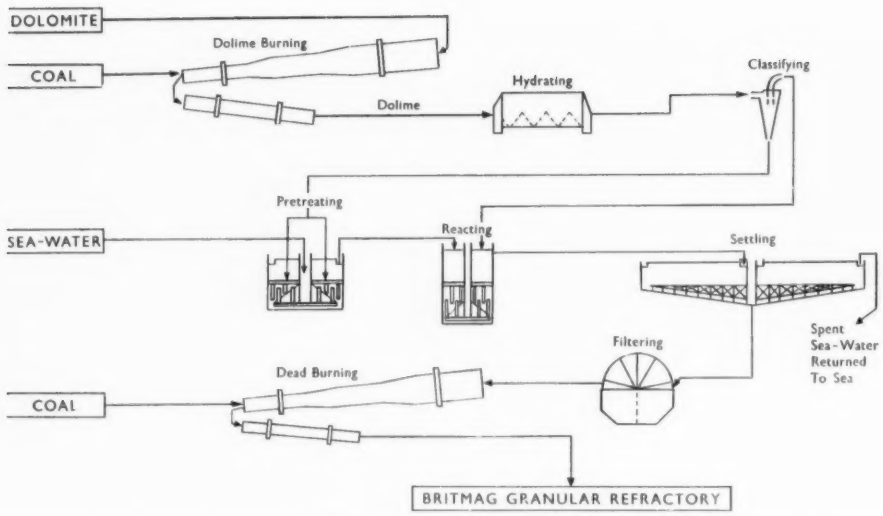
Quite apart from its strategic importance, the process is making a considerable contribution to the national prosperity, since the economics of the process are such that the Company is able to market magnesia at 70% of the import price, and to effect an annual saving in foreign exchange of £4 million. Moreover, independence from Austrian sources has freed the British brickmaker from the previously imposed export restrictions, with the result that magnesite and chrome-magnesite bricks to the value of £1 million a year are now being exported from this country.

On the technical side, sea water magnesite is superior to the mined product in at least one respect, in that its composition can be adjusted to meet the special requirements of the user.

Process and Plant

The raw materials for the sea water magnesia process are sea water, dolomite and coal, and this fact governed the selection of Hartlepool as the site for the extraction plant, since it is on the coast, close to dolomite deposits, and suitably located with respect to coalfields.

At the Company's Thrislington and Coxhoe quarries, graded dolomite is burned in kilns to a magnesian lime known as dolime. At Thrislington, the kiln is of the rotary type and has a length of 350 ft. It is fired with pulverised coal and equipped with a rotary cooler;



Diagrammatic flow sheet for the production of magnesia from sea-water

steel heat exchangers operate in the feed end of the kiln, and cooler. The calcining plant at Coxhoe includes a 300 ft. long, pulverised fuel-fired, rotary kiln equipped with a planetary cooler, and five 100 ft. high shaft kilns fired with coke and fitted with skip hoist feeds and automatic discharge mechanisms. All the kilns are lined with Britmag refractory bricks, insulated with firebricks in the burning zone, and are fitted with multi-cell high efficiency cyclones for dust collection.

Care is necessary in carrying out the calcination in that the product must contain a reactive form of magnesia, so that it will hydrate satisfactorily. In addition to the " stability " of the dolime, a watch must be kept on the CO_2 content, as it is desirable to avoid the presence of calcium carbonate, which would find its way into the final product—the burnt magnesia. Centralisation of the control equipment and instruments has been applied.

The dolime is transported in hopper-bottomed rail wagons to the Hartlepool plant, where it is slaked with fresh water to a fine dry powder in any of the five hydrator units. The hydrator units have self-regenerative heating systems, and are all fitted with automatic weight feeders. The hydrated product is made into a slurry for ease of handling and to facilitate mixing with sea water, and is then classified to remove the impurities introduced from the fuel during calcination of the dolomite. Two units have pneumatic and three units hydraulic classification plants.

Sea water is drawn by centrifugal pumps through 950 ft. long cast iron pipelines supported by three jetties constructed in greenheart timber. Three sea water pumphouses house five pumps operating at 25 ft. suction (atmospheric pressure is about 34 ft. of water) and driven by 600 h.p., 2,750 volt motors. The intake is controlled by one man from a central control panel, on which are indicated flow rates in other sections of the plant: so that close supervision of the process is possible. The pumps operate for about 7 hours on each tide, so that storage tanks for the sea water are essential. The total storage capacity of 8 million gallons is provided by four tanks, two constructed in pre-stressed concrete and two in orthodox reinforced concrete: the sea water enters the tanks by way of sand traps which allow the solids to settle.

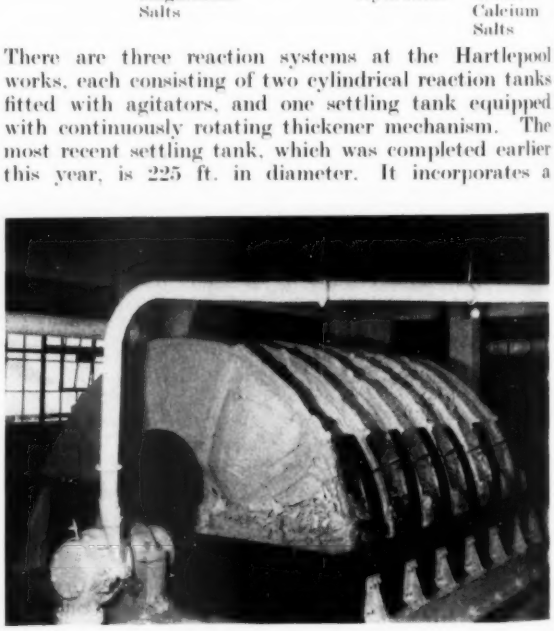
Before the reaction between the sea water and the dolime takes place, the former is treated to remove calcium bicarbonate, which would otherwise contaminate the magnesia later. For this purpose the storage tanks feed into five pre-treatment tanks or hydro-treaters, where a small proportion of dolime cream is added. The hydro-treaters are fitted with variable speed central drive and diaphragm sludge pumps.

After treatment, the sea water is pumped into reaction tanks, where it is mixed with the classified dolime slurry to precipitate magnesium hydroxide. The reaction is :—

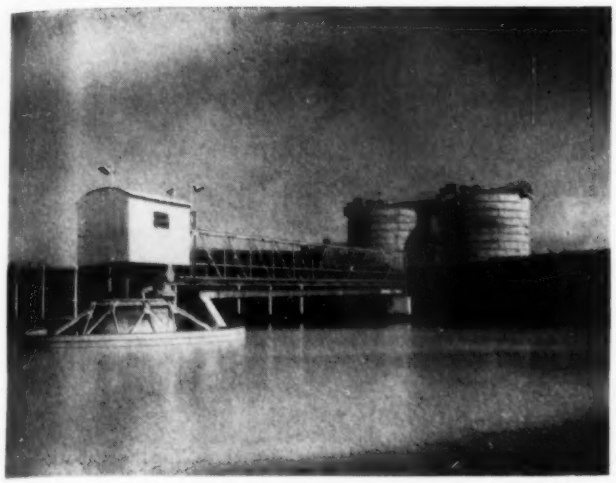
$$CaO\,MgO + \left\{\begin{matrix} MgSO_4 \\ MgCl_2 \end{matrix}\right\} + 2H_2O \rightarrow 2Mg(OH)_2 + \left\{\begin{matrix} CaSO_4 \\ CaCl_2 \end{matrix}\right\}$$

Dolime	Sea Water Magnesium Salts	Water	Magnesium Hydroxide	Spent Sea Water Calcium Salts

There are three reaction systems at the Hartlepool works, each consisting of two cylindrical reaction tanks fitted with agitators, and one settling tank equipped with continuously rotating thickener mechanism. The most recent settling tank, which was completed earlier this year, is 225 ft. in diameter. It incorporates a



Rotary disc vacuum filter.



General view of a reaction system, showing the two reaction tanks and the settling tank.

cantilevered thickening mechanism, is the largest central drive unit operating in Europe, and has a capacity of 3½ million gallons. Thirty million gallons of sea water are processed daily in the three reactor systems.

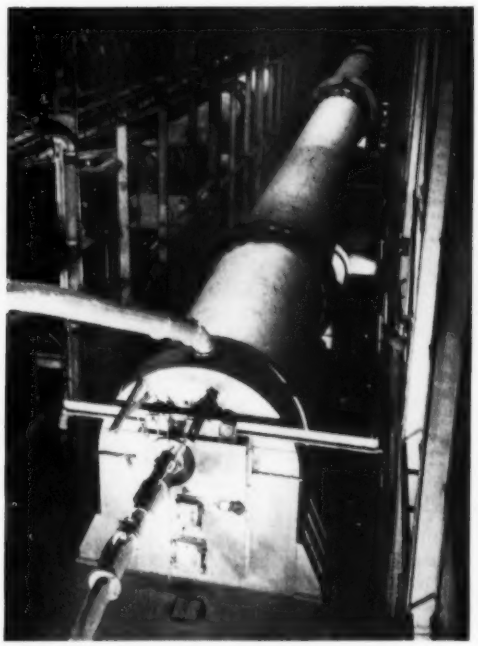
When the dilute suspension of magnesium hydroxide has settled in the tanks, it is drawn to a centre well by means of rotating rakes, and pumped out as a sludge to one of two systems containing sea water. Each system comprises one reaction tank and one settling tank with continuously rotating thickener mechanism. The washed sludge then passes to a battery of eleven rotary disc vacuum filters, with automatic pressure discharge gear and variable speed drive mechanisms. The filtering medium is heavy gauge staple nylon cotton spun fabric.

The filter cake, containing about 50% of solids, drops on to a moving conveyor belt which transfers it to the rotary kilns for calcination. There are five of these kilns, the largest being 250 ft. long, and each is equipped with an associated rotary cooler. The kilns, which operate at a temperature exceeding 1,600°C., are fired by pulverised coal and are fitted with waste heat regenerators. In the burning zone, they are lined with magnesite refractory bricks backed with firebrick insulation to minimise heat losses.

During its passage through the kiln, the sodium chloride content of the filter cake is volatilised and passes out with the hot gases, which also contain solid matter in suspension. Three 70 ft. high reinforced concrete spray towers provide counter current washing (with sea water) of the hot gases and these, working in series with three wet electric precipitators operating at 50,000 volts give a 99·5% clean gas discharge.

The Product and its Uses

After cooling, the granular product from the kiln—known as Britmag—is ready for bagging or dispatch in bulk to the brickworks and steelworks. It contains a minimum of 90% magnesium oxide (MgO), and is virtually inert to the action of atmospheric moisture. An average composition is: silica (SiO_2) 2·1%; alumina (Al_2O_3) 1·4%; iron oxide (Fe_2O_3) 1·4%; lime (CaO) 2·7%; and magnesia (MgO) 92·4%.



One of the five rotary kilns for producing granular Britmag.

Britmag is supplied from Hartlepool to Steetley's Refractory Brick Division and to other British refractory manufacturers. It finds its most extensive use in the production of magnesite and chrome-magnesite bricks, the latter containing about 30% of magnesia, which are used for the linings of open hearth steel melting furnaces. In granular form, it is employed as a fettling material on steel furnace hearths. Another important application is for the lining of cement and lime kilns, where the magnesite content of the refractory is raised to between 60 and 80% to meet conditions of severe abrasion.

In twenty-one years, sea water magnesia has evolved from the research laboratory to the full-scale commercial plant, an achievement which owes much to the work of the Steetley Company. During this period, the production rate per man hour has increased some sixty-fold, whilst the fuel consumption per ton of product has been quartered; at the same time the quality has improved in that the magnesia content is about 5% higher than it was in 1938. Current production capacity is 150,000 tons per annum of deadburnt magnesia, and the total production to date is of the order of a million tons. Though not now the only one of its kind in the world, the Hartlepool plant is still the largest, and it is playing a significant part in the national economy.

Refractory Mouldings Move

REFRACTORY MOULDINGS & CASTINGS LTD., of Kegworth, announce that owing to expansion of business they are moving to larger premises situated in Market Place, Kegworth. It is hoped that the move will result in an even more efficient service to customers, and particularly to those areas of Europe and the Far East to which increased quantities of products are being exported.

Recrystallisation of Tungsten Wires

By G. L. Davis, B.Sc., Ph.D., A.R.I.C., F.I.M.

Material Research Laboratory, Mullard Radio Valve Co. Ltd., Mitcham Junction

From observations of mechanical properties and microstructures, it was found that tungsten wires recrystallise firstly by formation of a columnar structure with good retention of strength and ductility. At higher temperatures, secondary recrystallisation occurs with embrittlement and lowered strength. The practically important temperature for onset of this coarsening is very sensitive to the nature of the starting material and its history. A hypothesis is advanced to explain the phenomena and the kinetics of the processes are considered.

(*Continued from page* 184 *of the October issue*)

Discussion of Results

Primary Recrystallisation

Primary recrystallisation appears to proceed in a normal manner, except that there is no change in orientation. The similarity of deformed and recrystallised textures could be explained in various ways, but, as nucleation was not observed, it would seem that this is a case of "recrystallisation *in situ*," as described by Beck[18] after Crussard. Growth of sub-grains which preceded recrystallisation in this process, could hardly be observed by optical microscopy on the fine fibres, although Delisle,[19] using electron microscopy, remarked the formation of sub-grain boundaries across fibres on annealing. His photographs on fine wires, indeed, show a widening of the fibres without nucleation on annealing. Perhaps the changes shown in Figs. 20 and 21 on swaged rod show a similar process on a larger scale.

This stage of recrystallisation comes to completion with the development of a columnar structure which may be remarkably stable: this is discussed further below.

Laurent and his co-workers,[20-22] in their excellent papers on the theory of recrystallisation, review the conclusions derived from the application of various analytical methods to the results of physical measurements in following recrystallisation processes. The several formulae derived by the differing theories can, as they say, be expressed in the general equation suggested by Avrami.[23, 24]

$$x = \text{Exp}\,(-Kt^a)$$

where x = fraction unrecrystallised

K = a temperature-dependent constant

$$= C \,.\, \text{Exp}\left(\frac{-Q}{RT}\right)$$

t = time

and a = a constant which must be determined experimentally for each process. Analytically, values of 1, 2 and 4 have been proposed, but, experimentally, it has been found to vary from 0·37–7·5.

The fraction recrystallised $(1-x)$ is best estimated by direct measurement, but in such cases as the present, where this cannot be done, an approximation is given by the change in some measured property[25]—in this case U.T.S. In the above expression:

$$K = \text{const. Exp}\left(\frac{-Q}{RT}\right)$$ — where Q is the activation energy of the processes considered—and thus Q can be calculated if either t or x is kept constant.

In the experimental section concerned with effect of

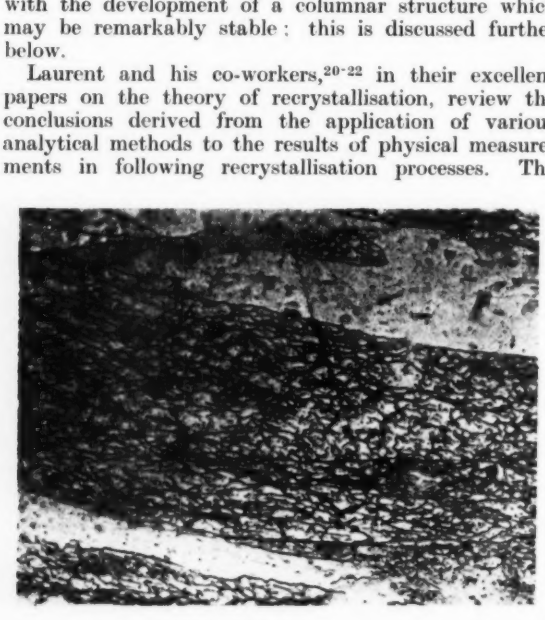


Fig. 20.—Microstructure of 6 mm. rod, annealed for 10 minutes at 1,300° C. × 150

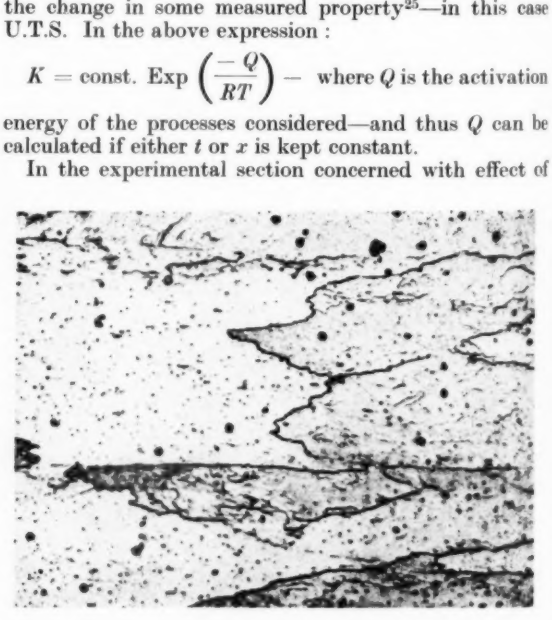


Fig. 21.—Microstructure of 6 mm. rod, annealed for 10 minutes at 1,500° C. × 150

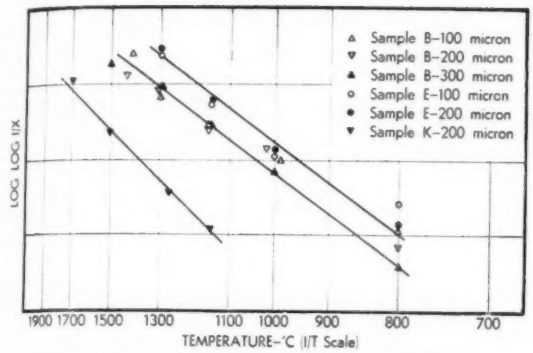


Fig. 22.—Kinetics of primary recrystallisation.

time of heating, a value of apparent activation energy was obtained for primary recrystallisation which should be modified by the factor α. The estimation of true activation energy from the graph gave 90 k. cal. $\times$ 0·28, i.e. 25 k. cal./gm. atom.

The relation given above can also be used to derive Q by plotting ln. ln. x against $1/T$ for constant time and estimating x from property changes. In this case any error due to the constant α is not involved in the calculation, but the uncertainty of the relationship used for determining x reduces the value of the method. Nevertheless several samples were examined in this way and the results are plotted in Fig. 22.

It may be seen that the activation energies (given by the slope of the lines) appear to be independent, within the limits of error, of the material, and its degree of deformation within the range examined. The value derived from these curves was *ca.* 25 k. cal./gm. atom, and this agrees well with that obtained above.

Secondary Recrystallisation

At some temperature in excess of that required for completion of primary recrystallisation, a second phase of softening occurs, associated with a renewed onset of grain growth. According as the nature and treatment of the material varies, there is a greater or lesser separation of the two steps in the strength curve, but, in most cases the overlap is so slight that it is remarkable that no reference to such steps have been reported elsewhere.

As in primary recrystallisation, coarsening of tungsten does not seem to occur by a normal nucleation process (*cf.* 16) but by coalescence of existing grains, and it is for this reason that the sensitivity to rate of heating shown in metals, in which the process of recrystallisation is initiated by nucleation, is not observed in wires.

Once initiated, secondary recrystallisation proceeds in one of two ways : (*a*) the formation of relatively small equiaxed grains which grow in a normal manner by consumption of each other ; (*b*) the formation of very large grains which grow by consumption of the preceding (columnar) structure directly. The first mode is exemplified by the behaviour of undoped or heavily doped or larger diameter wires, and is especially seen in samples which coarsen at low temperatures. Exaggerated grain growth occurs when coarsening is delayed to high temperatures.

In either case, the product is weak and brittle as compared to the columnar state. However, in those applications where secondary recrystallisation cannot be avoided, choice of materials to end with one of these two structures should be exercised. Fine-grained equiaxed material has a somewhat higher strength and resistance to mechanical shock, and is not subject to " offsetting." The coarse grained wires have good " sag " resistance suitable for lamp use.

If advantage can be taken of the strength, ductility and good forming quality of wires having a columnar structure, it is important practically, as well as interesting academically, to consider the manner of development of the structure and the reasons for its stability.

Development of a columnar structure during primary recrystallisation has frequently been observed—especially in wires after extensive deformation—and has been explained in several ways, but recrystallisation *in situ* from a fibrous structure would be expected to lead to such a structure, at least in the early stages of grain growth. It is less easily seen why the grain growth becomes restricted at a certain grain width and the boundaries do not further extend laterally, so that at this stage the process ceases. It is not until higher temperatures are reached that renewed activity sets in, and this is not of the same type as had led to the structure.

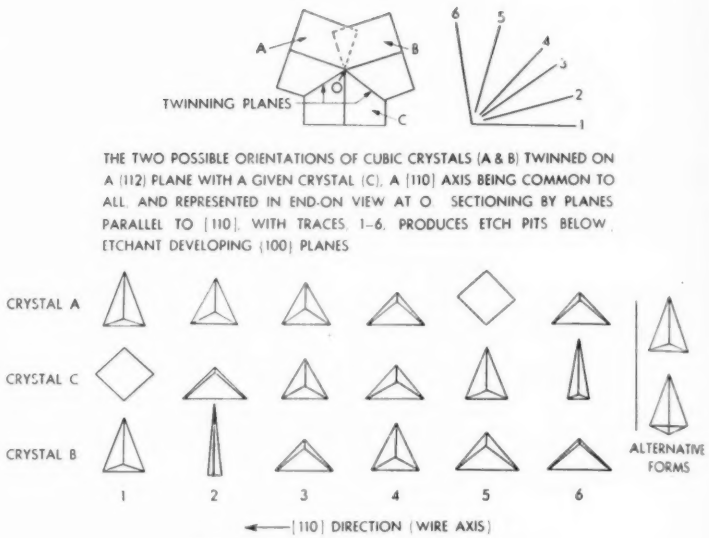
Thus, some restricting mechanism must be sought, in accord with the grain growth behaviour. Two suggest themselves : resistance to boundary movement by obstructing agents such as inclusions ; stability of the boundaries due to their being of low energy—the contiguous grains having a related orientation. A study of the results above indicate that neither explanation alone is sufficient, and it seems likely that high coarsening temperatures are a result of a combination of both factors. But for clarity of presentation, each will be considered, at first separately.

From the behaviour of " pure " wire as compared to those with suitable concentrations of additives, it is evident that (since textural effects can be presumed to be essentially similar) the presence of inclusions stabilises the columnar state. But it is clear from the results on those samples with rather high concentrations of inclusions that excessive amounts no longer act so effectively. This is partly due to the fact that many of the particles are too large (see ref. 26) and partly to the ease of formation of boundaries across the grains due to the presence of many inclusions within the grains. There is some evidence that improving the refractoriness of the additions helps to stabilise the boundaries.

It is certain that residual materials in extremely minute amounts have a profound effect on the behaviour of tungsten when it reaches the stage of fine wire. Two metal powders, apparently identical and treated in the same manner as regards doping and processing, but extracted from different ore supplies (themselves very similar in chemical composition), often have entirely different coarsening temperatures.[27] This fact is widely known in the tungsten wire industry.

Considering the nature of the refractory inclusions, the purer the refractory present in the metal, the higher and sharper, in general, will be its melting point. Since the amount of refractory is evidently small, the slightest trace of other material also present will have a profound effect on the recrystallisation behaviour. This is presumably the explanation of the great variations between wire produced from different ore sources—that traces of impurities are accidentally retained which are characteristic of the ore. (But note that Millner[11] considers accidental impurities to be of little importance).

Obstruction by micro-porosity, as suggested for sintered platinum,[28] could apply of course to tungsten but, in view of the sharp differences between batches having similar press densities, it would not appear to be the only, or even the chief, factor. Perhaps a more likely hypothesis would be to explain the behaviour of the platinum wires (apart from the purely textural effects) as due to minute amounts of impurities acting as inhibitors. The estimated metallic impurities in the platinum were quoted at very low values, e.g. 0·0001 to 0·005%, but these amounts converted into the corresponding volumes of oxides, some of which would not be reduced by hydrogen, represents amounts which cannot be neglected. If the effect from obstructors is removed, due to their solution, complete removal, or fusion and coalescence, coarsening will commence the more rapidly the greater the delay.[29]



THE TWO POSSIBLE ORIENTATIONS OF CUBIC CRYSTALS (A & B) TWINNED ON A (112) PLANE WITH A GIVEN CRYSTAL (C), A [110] AXIS BEING COMMON TO ALL, AND REPRESENTED IN END-ON VIEW AT O. SECTIONING BY PLANES PARALLEL TO [110], WITH TRACES 1–6, PRODUCES ETCH PITS BELOW, ETCHANT DEVELOPING {100} PLANES

Fig. 23.— Orientation of etchpits.

The inclusions can be expected to be arranged in the form of stringers, derived from their dispersion from grain boundaries at some earlier state of the history of the specimen. But consideration of the degree of deformation, together with the grain size at preliminary stages of manufacture, would suggest that there would be many more rows in a cross section than would account for the grain count in a typical columnar structure. Thus only some of the stringers are effective as inhibitors, which is unlikely without the intervention of some other stabilizing factor. A further need for such a factor arises since it has been shown that thermal and mechanical history also have influence on coarsening of a given material. This is most probably a textural cause, and it is possible that lateral grain boundary migration proceeds only so long as there is an appreciable difference of orientation between the growing grain and the grain being consumed. When two neighbouring grains have a nearly similar or twin relationship, however, the common grain boundaries would remain relatively stable. While this could operate for wires with only the need for a fibre texture, the restricted freedom of orientation of grains in sheet—uniquely orientated in a sample of high degree of preferred orientation—means that the grains would already have a low energy boundary. Recrystallisation *in situ* (without change of orientation) could then hardly lead to grain growth. It is therefore significant perhaps that in the recrystallisation of tungsten sheet with some departure from perfect texture, nucleation and growth are observed, but with a more nearly perfect texture, the driving force for grain boundary migration would be small indeed, and this would explain this resistance to recrystallisation of tungsten sheet showing a high degree of texture.

Evidence for the textural forces was sought in the study of etchpits in several hundreds of micrographs. Two types of low energy boundaries can be developed—with slight change in orientation and with twins; it is likely that the first type will be difficult to detect. Since tungsten twins on the (112) plane, it is possible to obtain two twins to a given grain all having a common [110] direction (parallel to the wire axis). This is illustrated in Fig. 23, and the etchpit patterns which would be produced by sectioning adjacent twins in a number of planes, are indicated—if the {100} planes are developed by the etch[30] (as was found to be the case). In almost every case examined, the etchpits conformed to these configurations. In a few cases, two grains of manifestly differing orientations were enclosed with a large grain. As Burgers[4] has shown, enclosed grains are usually twins, and thus two such could be found in a parent grain, all conforming to a [110] fibre texture.

It is, perhaps, significant too that a sample of platinum made by the powder method (kindly provided by one of the authors of the paper referred to[28]) was found to have the following textures :—

As received—deformation texture ..	[111] fibre axis
Recrystallised to columnar state ..	[111] traces of (110)
Coarsened	[110]

The retention of the [111] texture in recrystallised wire is rather unusual for face centred cubic metals, and it would seem justifiable to link this retention with the stability of the columnar state observed in this material and with tungsten wires.

It seems fair to conclude then that coarsening can be considered to be inhibited by a combination of textural effects and mechanical obstruction. However, both of these forces are subject to some measure of control: the amount, nature and distribution of the inclusions can be varied, and the degree of preferred orientation is dependent upon the deformation applied. In this way there is some possibility of supplying supporting evidence for the hypothesis, and some predictions can be made which may test its validity.

(1) In less perfect textures, a wider scatter in the preferred orientation will allow a coalescence more continuous and less abrupt with rising temperature. Coarsening can then be expected to commence at a lower temperature, to be more gradual, and to tend to produce more equiaxed structures.

(2) After annealing at temperatures above the fusion point of the refractory inclusions, much of the effect of the preceding working upon resistance to coarsening can be expected to be lost. Rising temperatures cannot be expected to have a discontinuous influence on effects due to insoluble impurities until their fusion point is

reached, although with mixed refractories this temperature may not be sharply defined.

(3) The behaviour, on heating, of a batch of material that has a high coarsening temperature will differ from that having a lower coarsening temperature. Inhibition from textural effects may be assumed to be similar for all batches, so that differences in the coarsening temperature between the two types must be ascribed to inhibition by the inclusions. The coarsening of the high temperature batch would be expected to show the following characteristics :—

(*a*) Reduced sensitivity to degree of deformation.

(*b*) Slight variation of coarsening temperature with the time of heating, since the insolubles will " melt " at their fusion temperature, even if this has been attained for the shortest times.

(*c*) Generally, the step in the tensile strength curve will be very marked, i.e. coarsening will set in very abruptly since, when the obstruction is removed by fusion, the high temperature at which this takes place will mean a high rate for structure change.

If the results for batches having a high coarsening temperature are studied, these predictions are found to be confirmed. Conversely, equiaxing may be expected to be favoured under the following conditions.

(*a*) Obstructions are absent—as in undoped wires.

(*b*) Dispersion, shape or size of inclusion are unfavourable—as in coarser wires with their lower degree of deformation and texture.

(*c*) Obstructors are present in excessive amounts, so that their size, number and close distribution within the grains will favour the formation of new boundaries.

(*d*) The refractory inclusions have a low melting point, so that when their obstruction fails, the columnar state is still stabilised to some extent by the texture.

(*e*) If the refractory is mixed and melts over a range of temperature, the coarsening temperature will not be sharply defined, and there will be a temperature dependent rate factor for the removal of obstruction.

From a consideration of the factors leading to equiaxing listed above, it may be inferred that all factors tending to lower the coarsening temperature would also tend to favour equiaxing. High coarsening temperatures would be mainly associated with direct coalescence of the elongated grains, to give a final structure of very large grains.

Conclusions

In tungsten wires drawn under controlled conditions, it was found that recrystallisation took place by a clearly marked two-stage process : (*a*) primary recrystallisation, and (*b*) coarsening, or coalescence, or secondary recrystallisation.

Many different processing and other variables were investigated for their influence on both stages of recrystallisation, and of those examined the following were shown to have the greatest importance on primary recrystallisation : temperature of working ; degree of deformation ; interstage annealing ; nature and amount of impurities ; time and temperature of heating.

The first stage of recrystallisation from the deformed state took place without change of preferred orientation to produce a structure of columnar grains, the wire remaining ductile. This structure appeared to be arrived at by a growth of fibres directly, without evidence of the usual nucleation and growth of more or less equiaxed new grains.

Secondary recrystallisation took place at a higher temperature than that at which primary recrystallisation appeared to have been completed, and was often extremely abrupt in its commencement. Again there was no change in preferred orientation.

The temperature of coarsening was found to be dependent on several factors, of which the most influential were : the nature and amount of insoluble inclusions ; the degree of deformation ; annealing at temperatures near the coarsening temperature followed by further deformations ; and time of heating.

The presence of small amounts of refractory oxides as inclusions favoured the stability of the columnar structure, and the higher the melting point of the oxide, the greater was its effectiveness. It was found, however, that excessive contents of such impurities reduced the stabilising effect, so that there is evidently an optimum amount.

It is suggested that both types of recrystallisation take place without a process of nucleation : recrystallisation is considered rather as a form of grain growth from existing grains or sections of grains. The progress of this change is arrested when neighbouring grains bear a simple orientation relationship, and a columnar structure is produced, which is stabilised further when the boundaries are aligned with strings of inclusions.

The columnar structure, formed by primary recrystallisation remains stable on further heating until disturbed in one of the following ways :—

(1) If the degree of deformation is insufficient to impart a nearly perfect texture and to disperse the impurities suitably, coarsening occurs immediately after, or overlapping primary recrystallisation.

(2) The inclusions presumed to be " locking " the grain boundaries melt, soften, dissolve, or are otherwise removed, and further grain boundary migration is facilitated.

(3) In the presence of an excessive quantity of refractory inclusions, many relatively large particles are present within the grains themselves and the columnar state is either never well established, or readily breaks down by the formation of low energy grain in boundaries across the grains.

As regards the mode of coarsening, two main types have been noted :—

(*a*) Equiaxing.

(*b*) Immediate transition from the columnar to the coarsened state.

Of these (*a*) occurs when coalescence takes place in the presence of excess inclusions ; or at low temperatures, as occurs when inclusions are absent or of low refractoriness ; or at low deformations ; or after long annealing times. The second mode (*b*) is favoured by those factors leading to high coarsening temperatures, e.g. high degree of cold working ; suitable amounts of a highly refractory additive, suitably dispersed ; short annealing times ; and the avoidance of high temperature interstage annealing.

Where the high melting point of the refractory inclusions is the most important factor in inhibition of

coarsening, the time of heating has little influence on the secondary recrystallisation temperature, but where textural inhibition is the main inhibitory agent, this temperature is dependent on heating time.

Acknowledgments

The author wishes to thank Dr. J. A. M. van Moll, Head of the Material Research Laboratory, and the Directors of the Mullard Radio Valve Co., Ltd., for permission to publish this work.

REFERENCES

18 Beck, P. A., *J. Met.*, 1952, **4**, 979.
19 Delisle, L., Davis, G. A., Physics of Powder Metallurgy, New York, 1951.
20 Laurent, P., *et al.*, *Rev. Met.*, 1949, **46**, 446.
21 Laurent, P., *et al.*, *ibid*, 1952, **49**, 485 and 593.
22 Laurent, P., *Met. et Corros.*, 1953, **337**, 333.
23 Avrami, M., *J. Chem. Phys.*, 1939, **7**, 1103.
24 Avrami, M., *ibid*, 1940, **8**, 212.
25 Balicki, M., Lewis, G., *J. Met.*, 1951, **3**, 981.
26 Burke, J. E., Atom Movements, 1950.
27 Nichols, M. H., *Phys. Rev.*, 1950, **78**, (2), 158.
28 Middleton, A. B., *et al.*, *J. Inst. Met.*, 1949, **75**, 595.
29 Talbot, J., *et al.*, *Comptes rend.*, 1953, **238**, 818.
30 Davis, G. L., *J. Scientific Instruments*, 1958, **35**, (4).

New and Revised British Standards

Blackheart Malleable Iron Castings (B.S. 310 : 1958) Price 4s.

Three grades of blackheart malleable iron castings (B18/6, B20/10 and B22/14) form the subject of this revision of a well-established British Standard last revised in 1947. The chemical composition of the metal of the castings is considered to be of less importance than the mechanical properties. In consequence, compliance with the requirements of the specified tests is to be regarded as the main criterion of the material's "suitability for purpose."

The provision and heat treatment of the test bars are specified, as are tensile test, bend test and hardness requirements; provision is also made for additional tests to be undertaken by arrangement with the manufacturer. Alternative dimensions—based on practice in several other countries—are given. They have been included in order to gain experience with them in the United Kingdom, and to facilitate the exchange of information with other countries.

Acid Resisting Silicon Iron Pipe and Pipe Fittings (B.S. 1333 : 1958) Price 6s.

This revised publication (22 pp. illustrated) deals with three types of pipes and pipe fittings: cone ended, spigot and socket, and plain ended. The size range is 1—12 in. nominal bore. The main differences between this and the first (1946) edition are (*a*) there are now requirements for loose coupling flanges; and (*b*) requirements for the 2½ in. (64 mm.) and 5 in. (127 mm.) sizes have been omitted.

The body of the specification contains clauses on material, workmanship, finish, testing and marking and (for the flanges) drilling and bolts and nuts. Each of the eight tables of standard dimensions is presented in association with a "keyed" diagram. Because of their high corrosion resistance, these pipes are widely used in the chemical industry. Hence the importance of the standard's statement that "the sockets of spigot and socket pipes will accept the plain spigots of chemical stoneware pipes of the same nominal bore."

An appendix to the publication lists the information which a purchaser should provide when submitting an enquiry or order; another contains a recommended method for coupling plain pipes.

Determination of Compressibility of Metal Powders (B.S. 3029 : 1958) Price 3s.

Compressed metal powders possess many unique properties, e.g., in their varied forms they contribute to the extreme hardness of tool steel, and to the essential porosity of certain types of bearings. Hence the timeliness of this short—but important—British Standard method for determining the compressibility of metal powders. The three clauses of this new publication specify: the die and set to be used (together with an illustration), the method of determining the weight of powder required, complete detail of the 'procedure' and the manner of expressing the result obtained.

General Recommendations for the Sampling of Manganese Ore (B.S. 3035 : 1958) Price 4s.

These recommendations are based on the work of Technical Committee 65 'Manganese Ore' of the International Organization for Standardization, and are almost identical with the general agreement reached at the ISO meeting in Leningrad in August, 1956. The 10-page publication relates to the sampling of manganese ore in freight wagons immediately before the ore is loaded into the ship or immediately after discharge at the port of arrival. It is applicable to all manganese ores. The recommendations concern the size of the ore, the certificated samples and the number of increments, method of sampling, and the reduction of the certificated sample. An appendix provides a recommended method for locating the points of sampling in the freight wagons.

Copies of these British Standards can be obtained from the British Standards Institution, 2, Park Street, London, W.1. (Postage will be charged extra to non-subscribers).

Annealing Furnace Order

The Incandescent Heat Co., Ltd., Smethwick, has recently been awarded a contract worth more than £500,000 for a large coil annealing installation at the new steelworks of the Sociedad Mixta Siderurgia Argentina (SOMISA). The order, obtained against strong continental competition, is for 25 single stack coil annealing furnaces with 72 bases, 32 forced cooling hoods, and 3 exothermic gas atmosphere plants, each rated at 10,000 cu. ft hr. The furnaces are built to the design of the Lee Wilson Engineering Co., Inc., of Cleveland, U.S.A., for whom Incandescent are manufacturing licensees. The plant will handle coils up to 60 in. diameter in stacks up to 168 in. high, and will anneal annually 300,000 tons of cold reduced sheet and tinplate. The equipment will be manufactured and completely assembled at the Smethwick works of The Incandescent Heat Co., Ltd., and the order will be completed within twelve months. Incandescent have already equipped major British and European steelworks with coil annealing furnaces, including the largest annealing plant in Europe at the Abbey Works of the Steel Company of Wales, Ltd.

Pressing and Sintering Characteristics of Certain Copper and Tin Powder Mixes

By G. R. Bell, B.Sc.(Eng.), A.R.S.M., A.R.I.C., A.I.M., F. B. Webb, A.M.I.Mech.E., and R. Woolfall

Developments in the control of the atomising process have permitted the manufacture of copper powders having apparent densities as low as 2·1 g./ml. The cost of manufacture of copper powder by atomising is lower than that of the conventional electrolytic processes. An investigation has therefore been made into the pressing and sintering properties of this type of powder in comparison with two electrolytic copper powders when admixed with 10% of tin powder. The results indicate that the atomised powder is an entirely satisfactory substitute for electrolytic powder and in some cases is preferable.

VIEWS have frequently been expressed in the past that an important obstacle to the development of powder metallurgy is the high cost of metal powders. In the specific case of copper powder—one of the most important of industrial powders—the most popular existing type of powder used for this purpose is that which is made by electrolysis. Electrolytic deposition is fundamentally an expensive process, and there are no obvious ways of substantially cheapening this powder below present day prices. Alternative methods of making suitable powders consist in reducing copper oxides or in manufacturing a substantially oxidised atomised powder which is then subsequently reduced in a reducing atmosphere. Both of these processes are, however, expensive, partly because of the high cost of the reducing atmosphere, and partly because of the high capital cost of the equipment involved.

As a method of comminution, probably the cheapest possible process is that of atomisation, in which the metal is melted and then atomised by some suitable design of jet using air, steam, water or other expansible fluid. However, molten copper atomised with either air or water develops a powder the particles of which are substantially spherical, and this leads to powders of high apparent densities—usually between 3·0 and to 4·0 g./ml. Powders of such high densities have poor pressing properties, exhibiting low green strength and requiring high pressures for any given apparent compact density.

In the manufacture of sintered porous bronze bearings, in which it is customary to press and sinter a mixture of 90% copper powder and 10% tin powder, it is necessary to use copper powder of a substantially lower apparent density, with better properties for pressing and facility for producing compacts of high and controlled porosity.

F. W. Berk & Co., Ltd., and Powder Metallurgy, Ltd., have for some time been carrying out experimental work with the object of producing a copper powder having a low apparent density by simple atomisation, and not requiring any expensive after treatments, such as oxidation or reduction. In the last year or so, very considerable flexibility and control has been achieved in metal atomisation processes and it has now become possible to make copper powders having highly irregular particle shapes with apparent densities as low as 2·1 g./ml. Although these powders are in the simple atomised condition, their physical appearance under the microscope is remarkably similar to that of the powder reduced from the oxide. Fig. 1 is a photomicrograph showing such an atomised copper powder (apparent density 2·5) under a magnification of 40 diameters.

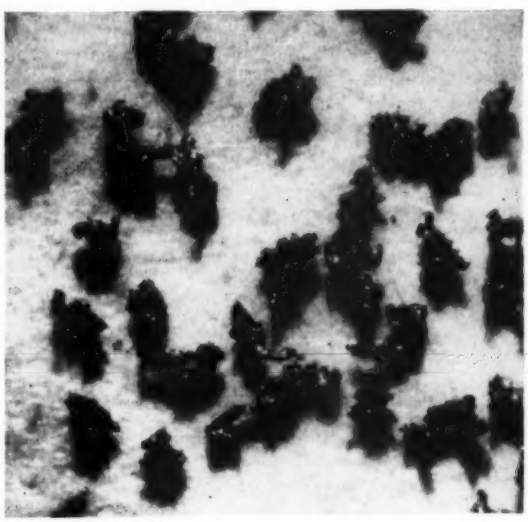


Fig. 1.—Photomicrograph of atomised copper powder NLD 2·5. ×40

In this paper the pressing and sintering characteristics of mixtures of some powders of this type with 10% of tin are reported in some detail, and the physical properties of the powders and of the pressed and sintered compacts are compared with some electrolytic powders and discussed.

Properties of the Powders

In the experimental work, comparison was made between: (*a*) two electrolytic copper powders of British origin, designated E1 and E2 and having a density of 2·6; and (*b*) three atomised copper powders having different apparent densities—2·3, 2·5 and 2·9—and one mixed powder comprising two parts of the powder of density 2·3 and one part of a powder of density 3·3.

These powders were designated NLD 2·3, NLD 2·5, NLD 2·9 and NLD Mix. The chemical analysis of these powders are set out in Table I and their physical proper-

TABLE I.—CHEMICAL ANALYSIS OF COPPER POWDERS

	NLD 2·3	NLD 2·5	NLD 2·9	NLD Mix	E1	E2
Cu : %	99·53	99·50	99·47	99·51	99·6	99·35
Pb : %	⩽0·02	0·04	0·02	⩽0·02	0·04	—
Sb : %	⩽0·02	0·02	⩽0·02	0·04	⩽0·02	⩽0·02
Loss in H_2 : %	0·08	0·11	0·12	0·13	0·13	0·16
Fe : %	0·03	0·05	0·05	0·02	—	—
S : %	⩽0·01	⩽0·01	⩽0·01	⩽0·01	—	—

ties, including apparent density, flow factor (Hall), tapped density, and mesh size distribution, in Table II.

The tin powder used for the experiments was air atomised by F. W. Berk & Co., Ltd., and is a typical tin powder employed commercially in Britain for the manufacture of porous bronze bearings : its chemical analysis and physical properties are detailed in Table III.

TABLE III.—PROPERTIES OF TIN POWDER

Chemical Analysis	Cu : % Pb : % Sb : % O_2 : % Fe : % Sn : %	0·26 0·45 0·09 0·15 ⩽0·02 Balance
Screen Analysis	+100 : % 100/200 : % 200/300 : % −300 : %	Trace 0·5 0·7 98·7
	Particle Size in Microns	Average % Present
Particle Size Analysis of −300 Mesh Fraction ..	⩽2 2–4 5–9 10–14 15–19 20–29 ⩾29	30 57 8·3 2·8 0·9 0·4 0·2
Flow Rate : sec.		Nil
Tapped Density : g. /ml.		4·5

Experimental Procedure

Blending

In each case copper and tin powders were taken in the proportion by weight of 90% of copper and 10% of tin, and the mixes blended for 60 minutes in a double-cone blender. In the cases of mixes incorporating lubricants, the metal powders were blended for 60 minutes, the relevant lubricant added as a dry powder, and the mix blended for a further 60 minutes, with the double-cone blender maintained at a temperature of approximately 55°C. The amount of lubricant added in such cases was $\frac{1}{2}$%. Table IV details the apparent densities, tapped densities, and flow factors (Hall) of the mixes.

Pressing

All pressings were made in a 190-ton upstroke press, and in every case the pressure employed was checked with a 100-ton proving ring just before pressing. The pressures recorded are believed to be accurate to ± 3%. Three separate die sets were used :—

(*a*) For the production of a 1 in. O.D. cylinder by pressing from the top only.

(*b*) For the production of a hollow cylinder, 1 in. O.D. and $\frac{3}{4}$ in. I.D., by pressing from both top and bottom by means of a spring-supported die body.

(*c*) For the production of tensile test pieces to B.S. 2590 by pressing from both top and bottom, as in the case of (*b*).

In the case of unlubricated powders, the internal surfaces of the die set and the external surfaces of the punches were lubricated with a mixture of powdered zinc stearate and carbon tetrachloride. This was carefully applied with a paint brush and allowed to dry before use.

Sintering

All sintering was carried out in a $2\frac{1}{2}$ in. I.D. silica tube heated externally by a resistance wound tube furnace. Temperature measurements were made by means of suitable thermocouples, placed internally in the tube in the centre of the compacts, and externally between the tube and the furnace wall, and heating and cooling cycles were recorded.

All sintering experiments were conducted in a mixture of three parts of hydrogen and one part of nitrogen, obtained ready mixed in cylinders from The British Oxygen Co., Ltd. Before entering the furnace, the gas was passed over platinised asbestos at 350°C. and dried with magnesium perchlorate. The gas flow was maintained at 2-2$\frac{1}{2}$ litres/min. throughout the sintering operation.

For sintering, all compacts were placed in a stainless steel boat, fitted with a lid, without any packing. The temperature of the central hot zone of the furnace was constant to ± 5°C. over the 5in. length containing the compacts.

Compact Testing Procedure

Density

The densities of all compacts were determined by measurement and weighing, with checks by lightly coating with cellulose lacquer and weighing in water.

Dimensional Changes

Dimensional changes were determined by radial and linear measurement.

Radial Crushing Strength

These tests were made in accordance with the procedure recommended by B.S. 1131.

Tensile Strength

A Hounsfield Tensometer was used for the determination of tensile strength by a procedure recommended by B.S. 2590.

TABLE II.—PHYSICAL ANALYSIS OF COPPER POWDERS

		Powder								
		NLD 2·3	NLD 2·5				NLD 2·9	NLD Mix	E1	E2
Nominal −300 Mesh Content		70%	65%	70%	75%	80%	70%	70%	50%	60%
Percentage Screen Analysis B.S.S.	+100 : %	Nil	Nil	Trace	Nil	Trace	Trace	Nil	0·2	0·1
	100/200 : %	20·3	14·9	18·2	9·4	8·5	14·9	16·6	36·0	22·1
	200/300 : %	12·0	18·4	10·3	13·9	9·7	14·0	15·1	13·8	16·1
	−300 : %	67·7	66·7	71·5	76·7	81·7	71·1	68·3	50·0	61·7
Fisher on −300 Mesh Fraction	High : μ	12·0	9·75	11·5	10·5	11·0	13·0	10·0	14·0	13·0
	Low : μ	12·0	9·25	11·5	10·5	11·0	12·5	10·0	14·0	12·5
Apparent Density (Hall) : g. /ml.		2·27	2·49	2·47	2·48	2·48	2·91	2·50	2·65	2·60
Flow Rate (50 g.) : sec.		35·4	34·6	34·0	36·4	38·7	30·0	32·9	25·0	27·8

TABLE IV.—PHYSICAL ANALYSIS OF COPPER + TIN MIXES

	Unlubricated Powders									Lubricated Powders			
	NLD 2·3	NLD 2·5				NLD 2·9	NLD Mix	E1	E2	NLD 2·5			
Nominal −300 Mesh Content	70%	65%	70%	75%	80%	70%	70%	50%	60%	70%			
Lubricant	—	—	—	—	—	—	—	—	—	Zinc Stearate	Lithium Stearate	Calcium Stearate	Stearic Acid
Apparent Density (Hall): g./ml.	2·58	2·67	2·71	2·69	2·68	3·16	2·72	2·76	2·71	3·23	3·22	3·35	3·26
Tapped Density: g./ml.	3·42	3·52	3·51	3·56	3·54	4·21	3·68	3·75	3·55	3·97	3·99	4·00	3·94
Flow Rate (50 g.): sec.	32·2	35·0	33·8	35·0	Nil	32·3	35·0	28·0	29·2	Nil	Nil	55	58

Elongation

The elongation was measured over a 1 in. central portion of the tensile test piece.

Hardness

Brinell hardness tests were carried out on the solid cylinders and on the ends of the tensile test pieces, using a 10mm. ball with a load of 500 kg. for 15 seconds.

Results

The results reported below may be divided into two main groups: (*a*) those on compacts made with unlubricated powders in a lubricated die set; and (*b*) those on compacts made in an unlubricated die set with powders to which solid lubricant had been added. All the results are presented in Figs. 2–22, with the exception of the elongation and hardness figures, which are quoted in Tables V–X. Every determination itemised on a graph represents an average of at least five separate observations.

Density

In Fig. 2 the apparent density of cold pressed compacts, expressed as a percentage of the theoretical density of the mixed metals, is plotted against the pressure used in producing the compact. It will be seen that, using log-log co-ordinates, the points lie on a straight line over the practical range of pressures 10×10^3 to 100×10^3 lb./sq. in. The densities obtained at any particular pressure with the atomised and electrolytic powders are almost identical, and the curve in Fig. 2 is almost identical with all.

Green Radial Crushing Strength

The radial crushing strength of the green compacts is plotted against the green density and the percentage green porosity in Fig. 3. These curves show the green strengths of the unlubricated powders, and it will be noted that NLD 2·3, NLD 2·5 and NLD Mix have substantially higher green strengths at all porosities than either of the electrolytic copper powders. NLD 2·9, on the other hand, shows a somewhat lower green strength than any other powder.

The curves in Fig. 4 show the green strengths at various green porosities of compacts made in unlubricated dies with NLD 2·5 and E1 powders when lubricated with stearic acid, zinc stearate, lithium stearate and calcium stearate. These results show that in all cases there is a substantial reduction in green strength caused by the addition of lubricant, and that compacts lubricated with

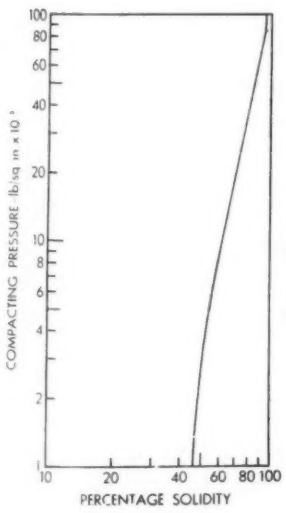


Fig. 2.—Curve showing the percentage solidity of copper-tin compacts made at varying pressures.

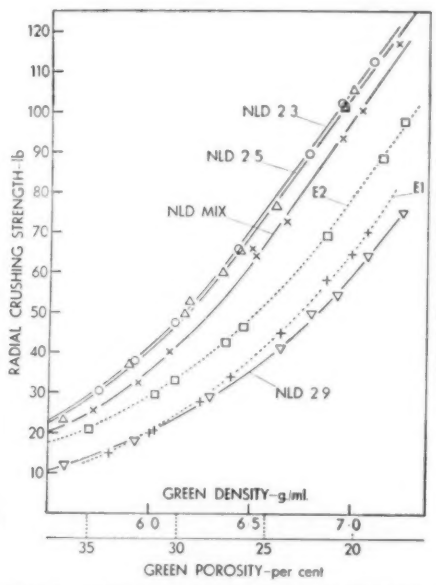


Fig. 3.—Curves showing the radial crushing strength of green copper-tin compacts, made with unlubricated powders in lubricated moulds, at varying densities.

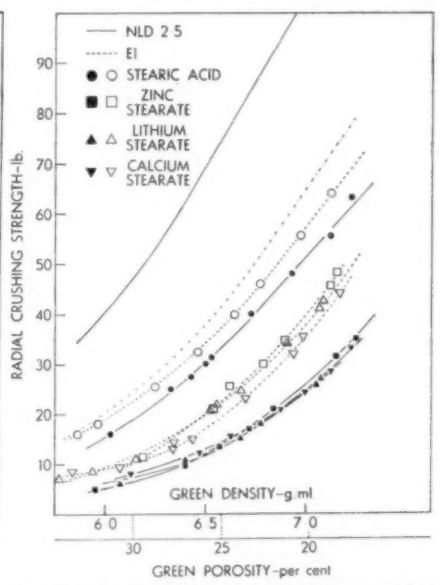


Fig. 4.—Curves showing the radial crushing strength of green copper-tin compacts, made in unlubricated moulds with NLD 2·5 and E1 powders lubricated by various materials. The curves for the same powders without added lubricant are shown for comparison.

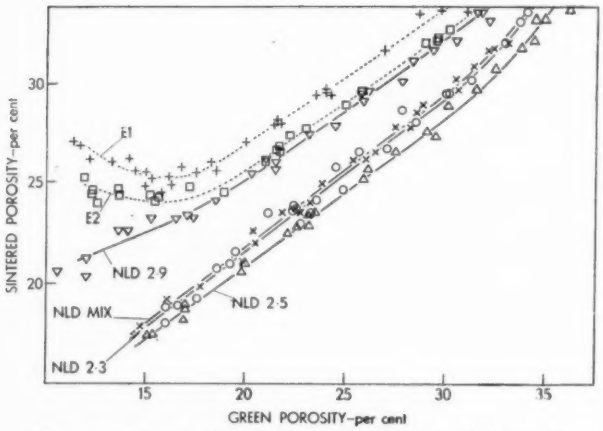


Fig. 5.—Curves showing the relationship between the porosities before and after sintering of copper-tin compacts made with unlubricated powders.

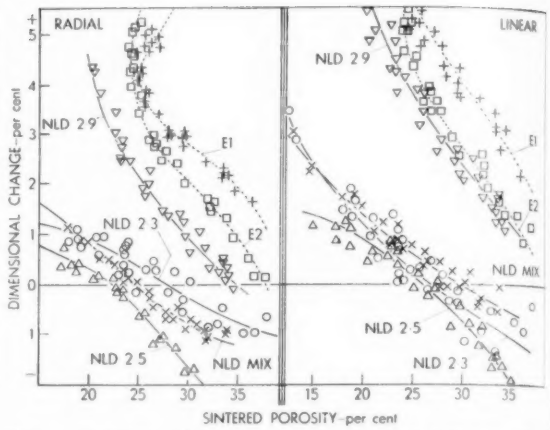


Fig. 6.—Curves showing the relationship between the dimensional changes on sintering and the sintered porosity of copper-tin compacts made with unlubricated powders.

metallic stearates show substantially lower green strengths than those lubricated with stearic acid. In each case the reduction in strength is greater with the atomised powder than the electrolytic, although the green strength at all densities is higher in the case of the atomised powder lubricated with stearic acid than in the case of the electrolytic powder lubricated with the metallic stearate.

Properties of Sintered Compacts

The curves shown in Figs. 5-8 refer to compacts which have been sintered at 825°C. for ten minutes, and Fig. 9 shows a typical time-temperature cycle for this operation.

In Fig. 5, which concerns sintered unlubricated powder compacts, the sintered porosity is plotted against the green porosity, and the curves indicate, therefore, the change in bulk porosity of the compact on sintering. It will be clear from the curves that each of the electrolytic copper powders exhibits a substantially larger growth on sintering than any of the NLD range of atomised powders. Of the latter, NLD 2·5 shows the smallest growth and NLD 2·9 the largest. Both the electrolytic powders have a point of minimum sintered porosity at the low green porosity end of the graph, thus making it impossible to produce a sintered compact having a bulk porosity of less than 24%.

The radial and longitudinal changes in dimensions on sintering are plotted in Fig. 6 in relation to the porosity of the sintered compact. The determinations were made on hollow cylinder pressings, and the changes expressed as percentages of the original dimensions. The curves indicate both growths and shrinkages.

The left-hand side of Fig. 6 shows clearly the high radial growths of the electrolytic powder compacts, indicating the minimum sintered porosity that can be obtained. With the exception of NLD 2·9, the other NLD powders show that either a growth or a shrinkage can be obtained, as desired, according to the initial cold pressing porosity. Thus, for example, with NLD 2·3, NLD 2·5 and NLD

TABLE V—EFFECT OF LUBRICANTS ON HARDNESS.

Porosity	Brinell Hardness: 10 mm. ball, 500 kg., 15 sec.									
	Unlubricated Powders						NLD 2·5 + Lubricant			
	NLD 2·3	NLD 2·5	NLD 2·9	NLD Mix	E1	E2	Zinc Stearate	Lithium Stearate	Calcium Stearate	Stearic Acid
15%	—	53·4	—	49·0	—	—	45	45	40·7	42·4
20%	47·5	43·4	—	42·0	—	—	39·7	40·4	39·0	39·6
25%	40·2	38·1	37·5	33·1	42·0	32·2	34·1	35·6	35·1	36·0
30%	34·4	34·4	32·0	27·3	32·5	26·8	28·6	30·6	29·5	30·5
35%	26·0	25·0	26·0	24·0	26·7	23·8	23·0	26·0	24·5	22·7
40%	21·1	20·9	20·8	21·8	21·8	21·1	19·7	21·3	19·4	18·6

TABLE VI.—EFFECT OF LUBRICANTS ON ELONGATION.

Porosity	Elongation: % on 1 in. gauge length									
	Unlubricated Powders						NLD 2·5 + Lubricant			
	NLD 2·3	NLD 2·5	NLD 2·9	NLD Mix	E1	E2	Zinc Stearate	Lithium Stearate	Calcium Stearate	Stearic Acid
15%	5	6½	—	5	—	—	4½	8	1½	4½
20%	3	4	—	4½	—	—	3½	8	1½	4
25%	2	3½	7½	3	12½	9½	3	7½	1½	3
30%	2	3	5	2½	11½	8	2½	6	1½	2
35%	2	2	4½	2	9½	7½	1½	5½	1½	2
40%	1½	2	3	2	5	4½	1½	5	1½	2

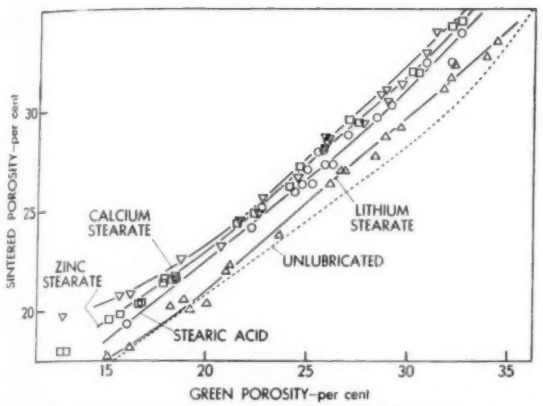


Fig. 7. Curves showing the relationship between the porosities before and after sintering of copper-tin compacts made with NLD 2·5 powder, with and without added lubricant.

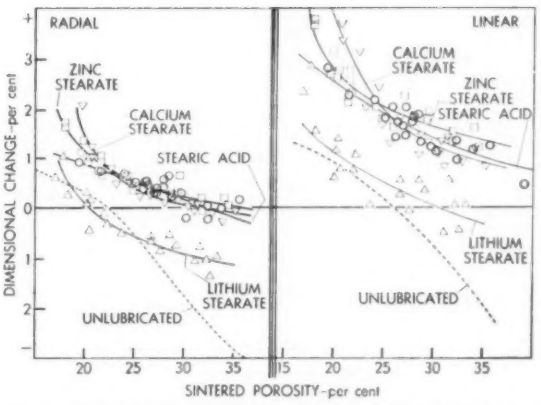


Fig. 8.—Curves showing the relationship between the dimensional changes on sintering and the sintered porosity of copper-tin compacts made with NLD 2·5 powder, with and without added lubricant.

Mix, growths are obtained at a sintered porosity of 20%, and shrinkages with a figure as high as 30%. As may be seen from the right-hand section of Fig. 6, the longitudinal dimensional changes follow a similar pattern, in that E1, E2 and NLD 2·9 powders exhibit high growths, whilst the remaining NLD powders exhibit either growth or shrinkage according to the initial porosity.

The effect of added lubricants is shown in Figs. 7 and 8. The former, in which the sintered porosity is plotted against the green porosity for the NLD 2·5 powder—with and without added lubricants—shows that the addition of powdered lubricant substantially increases the growth during sintering, except in the case of lithium stearate, which has a negligible effect. Fig. 8 shows that the radial and longitudinal dimensional changes resulting from the addition of stearic acid, zinc stearate and calcium stearate to NLD 2·5 indicate, in the main, growth, whereas the lithium stearate lubricated powder may exhibit either a growth or a shrinkage, as in the case of the unlubricated powder.

Sintered Radial Crushing Strength

In Fig. 10, the radial crushing strength, expressed as a percentage of the M.P.A. constant $K(=22{,}500)$, is plotted against sintered porosity for a series of sintered copper-tin cylinders made from the range of powders. The results indicate that with more than 28% sintered porosity, compacts made with atomised powders have a higher radial crushing strength than those made from electrolytic powders. Below 28% porosity, the electrolytic powders tend to show the better figures, but as it is impossible with these powders to obtain a porosity below 24%, comparison at lower porosity figures is impossible. It will be noted that with less than 29% sintered porosity, all the powders are higher than the M.P.A. recommended minimum. Of all the powders, NLD 2·9 has the highest sintered radial crushing strength.

Fig. 11 shows the effect of the addition of powdered lubricants on the radial crushing strength of sintered copper-tin cylinders made with NLD 2·5 powder. It will be observed that, in comparison with the unlubricated powder, each lubricant added reduces the radial crushing strength substantially, and that calcium stearate has the greatest effect in this respect. With the exception of that for the calcium stearate lubricated compact, the curves show that all the radial crushing strengths are greater than the M.P.A. minimum at less than 26% sintered porosity.

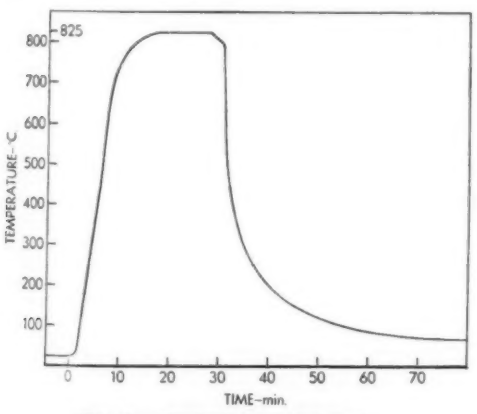


Fig. 9.—Typical sintering cycle.

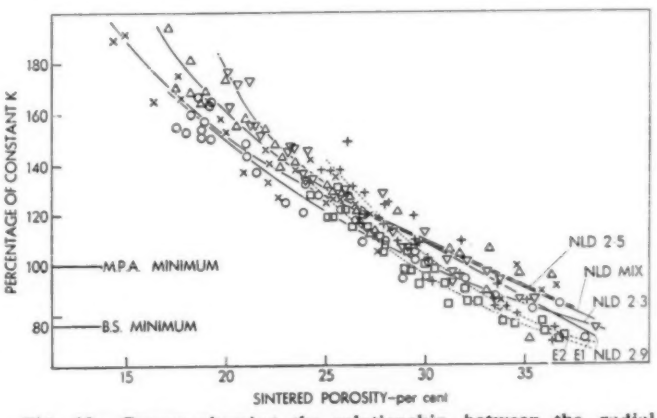


Fig. 10.—Curves showing the relationship between the radial crushing strength (expressed as a percentage of the M.P.A. constant *K*) and the sintered porosity of copper-tin cylinders made with unlubricated powders.

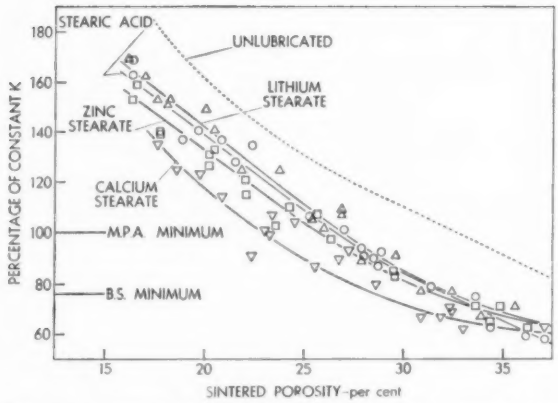


Fig. 11.—Curves showing the relationship between the radial crushing strength (expressed as a percentage of the M.P.A. constant *K*) and the sintered porosity of copper-tin cylinders made with NLD 2·5 powder, with and without added lubricant.

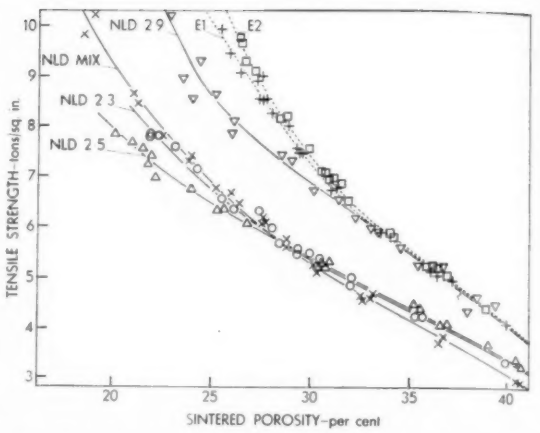


Fig. 12.—Curves showing the relationship between the tensile strength and the sintered porosity of copper-tin test pieces made with unlubricated powders.

Tensile Strength

In Fig. 12, tensile strength is plotted against sintered porosity for test pieces made with the range of powders, without added lubricant. It will be noted that those made with electrolytic powders show substantially higher tensile strengths than the rest, with the exception of NLD 2·9 test pieces, whose strengths approach those for E1 and E2.

The effect of adding powdered lubricants to NLD 2·5 powder is shown in Fig. 13, from which it will be seen that the addition of stearic acid or lithium stearate results in no significant change in tensile strength, except at lower porosities, when there appears to be some improvement. The addition of calcium stearate substantially lowers the sintered tensile strength.

Variation in Powder Mesh

As the atomising process developed for the production of the copper powder affords considerable facility for variation of mesh size without substantial change in the cost of manufacture, some experimental work was carried out to ascertain the influence of varying the proportion of −300 B.S. mesh material in the powders.

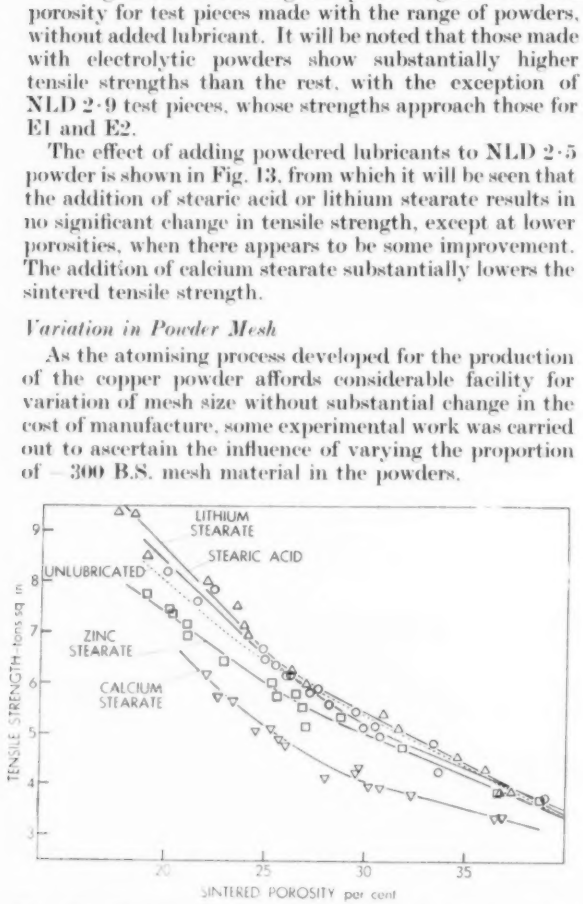


Fig. 13. Curves showing the relationship between the tensile strength and the sintered porosity of copper-tin test pieces made with NLD 2·5 powder, with and without added lubricant.

In Fig. 14, the radial crushing strengths of cold compacted hollow cylinders of NLD 2·5 powders containing, respectively, 65%, 70%, 75% and 80% of −300 mesh material, are plotted against green porosity. The curves show that coarsening the powder slightly increases the green strength of the compacts. The sintered porosity is plotted against the green porosity in Fig. 15 for the same range of powders. It will be seen that the coarser powders exhibit slightly higher growths, and that all the curves are parallel. Fig. 16 shows the dimensional changes on sintering, radial changes on the left, and longitudinal (i.e. in the direction of pressing) on the right. It will be seen that, as before, either growth or shrinkage can be obtained, according to the green porosity. The coarsest powder exhibits the largest growth, and the

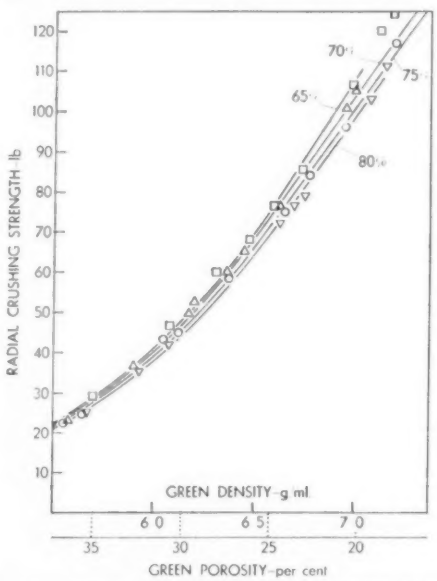


Fig. 14.—Curves showing the relationship between the radial crushing strength and the green porosity of cold compacted copper-tin hollow cylinders made with NLD 2·5 powder containing various percentages of −300 mesh material.

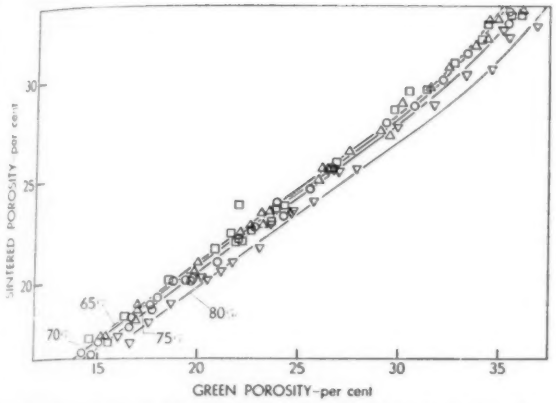


Fig. 15.—Curves showing the relationship between the porosities before and after sintering of copper-tin cylinders made with NLD 2·5 powder containing various percentages of −300 mesh material.

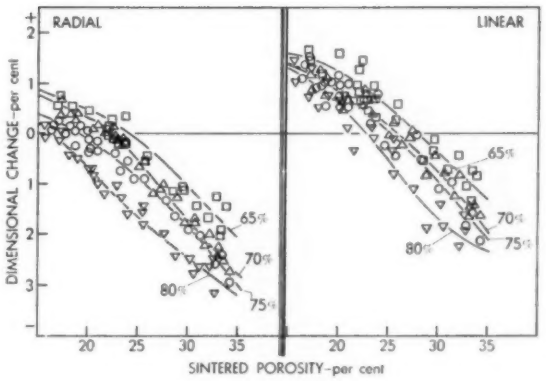


Fig. 16.—Curves showing the relationship between the dimensional changes on sintering and the sintered porosity of copper-tin compacts made with NLD 2·5 powder containing various percentages of −300 mesh material.

finest the largest shrinkage, both radially and longitudinally.

The influence of variation of −300 mesh content on the radial crushing strength of sintered cylinders is shown in Fig. 17, from which it will be seen that the finest powders have the highest strengths, particularly at low porosities. The curves for tensile strength in Fig. 18 show similar features.

In Table VII and VIII are recorded the variations in Brinell hardness and elongation with variation in the − 300 mesh content. The results indicate that no substantial change is brought about by variation in particle size in the range considered.

Variation in Time and Temperature of Sintering

As it is doubtful whether any two sintered bronze manufacturers sinter at precisely the same temperature, or for precisely the same time, experiments were made to ascertain the influence of such variables. Using NLD 2·5 copper powder and sintering the copper-tin mix at

TABLE VII.—EFFECT OF GRAIN SIZE ON HARDNESS.

Porosity	Brinell Hardness: 10 mm. ball, 500 kg., 15 sec. —300 Mesh Content of the Copper Powder			
	65%	70%	75%	80%
15%	51·5	53·4	50·3	50·3
20%	44·9	43·4	44·9	47·5
25%	38·1	38·1	37·7	37·3
30%	31·2	34·4	30·3	29·8
35%	25·9	25·0	27·1	25·5
40%	22·8	20·9	22·8	21·8

TABLE VIII.—EFFECT OF GRAIN SIZE ON ELONGATION.

Porosity	Elongation: % on 1 in. gauge length —300 Mesh Content of the Copper Powder			
	65%	70%	75%	80%
15%	7	6½	7	8
20%	3½	4	4½	4½
25%	2½	3½	3	3½
30%	2	3	2½	3
35%	1½	2	2	2
40%	1½	2	1½	2

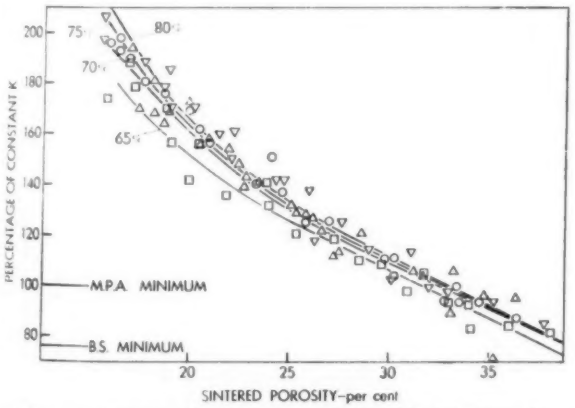


Fig. 17.—Curves showing the relationship between the radial crushing strength (expressed as a percentage of the M.P.A. constant K) and the sintered porosity of copper-tin cylinders, made with NLD 2·5 powder containing various percentages of −300 mesh material.

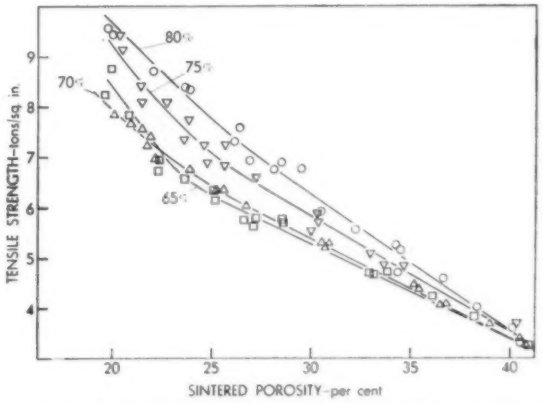


Fig. 18.—Curves showing the relationship between the tensile strength and the sintered porosity of copper-tin test pieces made with NLD 2·5 powder containing various percentages of −300 mesh material.

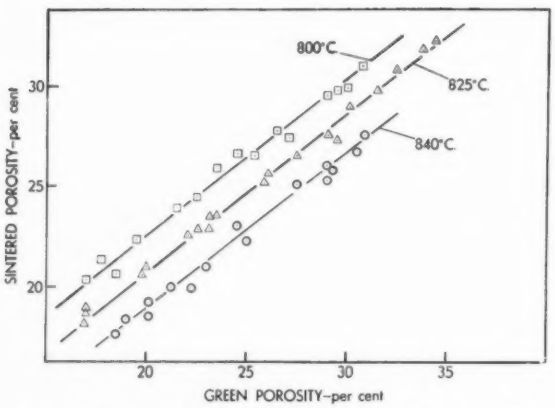


Fig. 19.—Curves showing the relationship between the porosities before and after sintering of copper-tin compacts made with NLD 2·5 powder and sintered at various temperatures.

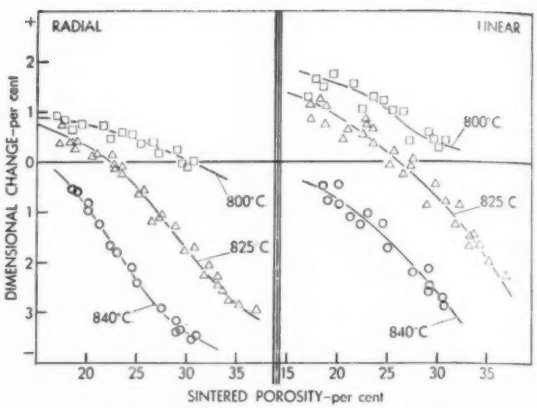


Fig. 20.—Curves showing the relationship between the dimensional changes on sintering with the sintered porosity of copper-tin compacts made with NLD 2·5 powder and sintered at various temperatures.

825°C., variation in the time of sintering over the range 5–30 minutes resulted in no substantial changes in growth, strength or hardness, thus indicating that the atomised powders are in no way sensitive to time of sintering.

The curves in Fig. 19, where sintered porosity is plotted against green porosity for copper-tin compacts made with NLD 2·5 powder and sintered at 800°C., 825°C., and 840°C., show that the growth is substantially diminished as the temperature is increased over this range. The compacts are very sensitive to sintering temperature, as in the case of electrolytic copper powders, and temperature variation thus affords an admirable means of varying the growth-shrinkage characteristics.

Variations of radial and longitudinal dimensional changes with sintering temperatures are shown in Fig.20, from which it will be seen that with a sintering temperature of 800°C. radial growth only occurs with porosities less than 30%, whilst substantial radial shrinkages occur with sintering temperatures of 825°C. and 840°C. Similar results are observed with the longitudinal changes.

Fig. 21 shows the influence of sintering temperature on crushing strength, namely that increasing the sintering temperature produces substantial reductions in the sintered crushing strength, but the effect on tensile strength is negligible, as may be seen from Fig. 22.

The figures in Tables IX and X show that increasing the sintering temperature causes substantial increases in both Brinell hardness and elongation in the tensile test.

Discussion of Results

A consideration of the results detailed above indicates that a suitable atomised copper powder of low apparent

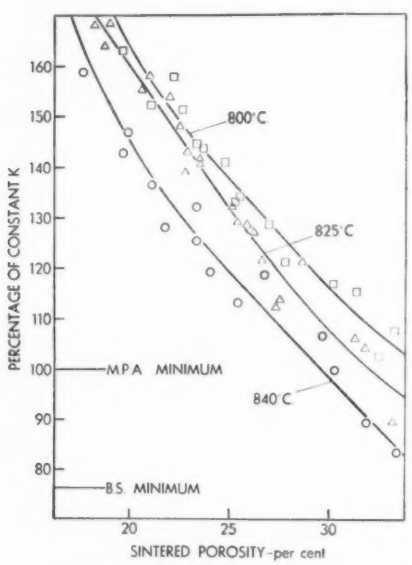


Fig. 21.—Curves showing the relationship between the radial crushing strength (expressed as a percentage of the M.P.A. constant K) and the sintered porosity of copper-tin cylinders made with NLD 2·5 powder and sintered at various temperatures.

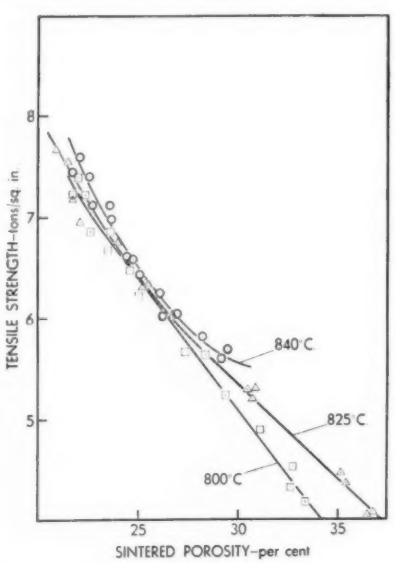


Fig. 22.—Curves showing the relationship between the tensile strength and the sintered porosity of copper-tin test pieces made with NLD 2·5 powder and sintered at various temperatures.

TABLE IX.—EFFECT OF SINTERING TEMPERATURE ON HARDNESS.

Porosity	Brinell Hardness: 10 mm. ball; 500 kg.; 15 sec. Sintering Temperature		
	800° C.	825° C.	840° C.
20%	40·2	43·4	47·5
25%	36·0	38·1	38·1
30%	31·2	34·4	35·4

TABLE X.—EFFECT OF SINTERING TEMPERATURE ON ELONGATION

Porosity	Elongation: % on 1 in. gauge length Sintering Temperature		
	800° C.	825° C.	840° C.
20%	3¼	4	7
25%	3½	3½	5½
30%	2½	3	4

density, when admixed with tin, can imitate to a remarkable extent the pressing and sintering characteristics of an electrolytic copper powder, although the cost of manufacture of such atomised powder is substantially lower.

In detail the results show, as compared with the electrolytic powder :—

(*i*) Almost identical pressure/density relations.

(*ii*) Substantially higher green strengths with unlubricated powders.

(*iii*) Lower green strengths with lubricated powders, although the difference is not substantial when using stearic acid.

(*iv*) Dimensional changes indicate lower growths than electrolytic copper powder, but the atomised powder has the advantage that either growth or shrinkage can be obtained.

(*v*) A considerable control over growth or shrinkage can be obtained by a variation in sintering temperature.

(*vi*) Some control over dimensional changes can be obtained by varying the mesh size of the atomised powder.

(*vii*) Sintered crushing strengths are very similar to those of electrolytic copper powder.

(*viii*) Tensile strength figures are somewhat inferior to those of electrolytic copper powder.

(*ix*) Elongation per cent figures are substantially lower with atomised powder than with electrolytic, although not sensibly inferior provided lithium stearate is used as a lubricant.

It would appear that lithium stearate is the best all round lubricant addition for this type of atomised copper powder unless good green strengths are desired, in which case stearic acid is preferable.

Conclusion

From a study of the results of this investigation, it would appear that atomised copper powder of this type affords an entirely satisfactory substitute for typical electrolytic copper powders—in a number of cases it is preferable—at a lower manufacturing cost. There is one feature of these atomised powders which is substantially an improvement over electrolytic powders, namely the keeping properties on storage. Electrolytic copper powders, unless protected by chemical additions, tend to oxidise very quickly, whereas atomised powders of this type are extraordinarily stable and preserve their original oxide content in open containers for many years.

Hydraulic Steel Tube Extrusion Press

THE first hydraulic extrusion press to be built in Germany for the extrusion of steel tubes and sections, has been operating for some months. The makers, Schloemann AG., Dusseldorf, are at present erecting in Japan a further plant of this type, which in addition to the 1,600/1,800 ton extrusion press with run-out trough, includes a 630 ton piercing press, a cooling bed, a 160 ton stretcher, and a pressure water station.

The press will produce tubes as well as solid and hollow sections of plain and alloyed steels. Starting billets have a diameter of 160 to 200 mm. (6·1 to 7·9 in.) at a maximum length of 800 mm. (31·5 in.) and will be extruded at 1,100 to 1,250° C. Operation is according to the Séjournet glass lubricated process in order to prevent scale, to insulate from heat and to reduce friction. The time of contact between material and tools during extrusion must be kept as short as possible so as to obtain sufficient strength and life of the mandrels and dies. Consequently the delivery speed of extrusion is very high. The extruded stock is sent to a stretcher where it is untwisted and straightened. This also results in removal of the vitreous skin which may still be found on the material.

The press can be fully automatically controlled in five programmed sequences, or electrically by hand as individual control. The automatic control permits the client to obtain an extrusion sequence of 45 to 60 seconds in accordance with the intended production programme. This means an output of 60 to 80 extrusions per hour.

Stordy-Hauck Oil Burning Equipment

STORDY ENGINEERING, LTD., announce that they are now able to provide to industry the complete range of the genuine Hauck designed fully proportioning oil burner, together with the special ancillary equipment which has given Hauck equipment a well deserved world-wide reputation for reliability, accuracy of control, etc.

The Stordy Organisation contains competent technical engineers who are willing to discuss any combustion problems where it is probable that Hauck equipment can be used with success, and it holds the licence for manufacturing the complete range of Hauck combustion equipment including the combined gas/oil burner. This includes a micro cam oil valve, self-clean metering oil valve, a proportioning oil burner, combination proportioning oil and gas burners, radiant tube burners (for either oil or gas), and radiant tube gas burners.

PROTOLITE, LTD., announce the transfer of their Main Office from Central House, Upper Woburn Place, London W.C.1, to the premises of the Parent Company, Murex, Ltd., at Rainham, Essex. The address is now: Protolite, Ltd., Rainham, Essex. Telephone: Rainham, Essex 3322. Telegrams: Protolite, Rainham-Dagenham Telex.

Coil Spring Forum

IN opening the Technical Forum on Springs and Spring Material, arranged by the Coil Spring Federation Research Organisation and held at the Wellington Hotel, Rochdale, on September 24th, the President, MR. R. SALTER BACHE said: "Research is not a pastime, it costs money and it involves work; the only justification for research is its value. We believe that there are a number of aspects of spring manufacture, in materials, processes and properties, about which we know very little. Our Organisation is designed to provide new knowledge, but new knowledge is not of any value unless it is utilised."

Application of Research Results

Continuing, Mr. Bache said that it was up to the industry to implement research information by putting it into practical use. This meant that the research results must be understood, and it was important that each firm should have staff available who could test and apply the research results in their own workshops.

Mr. Bache emphasised that spring makers should be experts in the products they make. They should be alive to all modern developments. Although his comments had dealt primarily with research in relation to spring manufacturers, there was no doubt that because of the universal use of springs, any developments which the C.S.F.R.O. might bring forward, were of importance to the engineering industry generally, and consequently of interest to a far wider field than just the manufacturers of springs. It was of the greatest importance that manufacturers should support technical development in their own industry, and also that they should benefit from the Spring Research Organisation.

MR. R. HAYNES, the Director of Research, who was acting as Chairman, introduced the Members of the Panel: MR. J. K. BACHE (Technical Director, Geo. Salter & Co. Ltd., West Bromwich); MR. F. GARTSIDE (Technical Manager, The Tempered Spring Co. Ltd., Sheffield); MR. J. A. ROBERTS (Technical Manager, Herbert Terry & Sons Ltd., Redditch); and MR. R. S. ROPER (Chief Metallurgist, F. S. Ratcliffe (Rochdale) Ltd., Rochdale).

Titanium Springs

The first topic discussed concerned the use of titanium as a spring material, particularly in comparison with stainless steel and in relation to its low modulus of elasticity. The chief advantage was the extreme resistance to corrosion of materials such as the heat treated I.C.I. 314A alloy compared with austenitic-stainless steels: the modulus of rigidity of this alloy—$9{\cdot}2 \times 10^6$ lb./sq. in.—was of the same order as that of the austenitic steels. The figure for hard drawn 318A alloy was in the region of 6×10^6 lb./sq. in. These lower modulus figures, as compared with steel, would affect the natural frequency of the spring, as would the lower density, and although a saving in weight was obtained by reducing the number of coils and by the lower density, due to the latter, one would have to increase the wire size to combat the lower natural frequency. Mr. Haynes said that a design study on titanium for springs was being undertaken in order to make a comparison with stainless and other steel springs, and the results of this investigation would be issued to members of C.S.F.R.O. in due course.

The panel was asked whether there were any new developments in wire drawing lubricants for both carbon and stainless steel which could be applied for automatic spring coiling machines: stainless steel was more difficult to coil on automatic machines and some spring makers ordered diamond drawn stainless steel with a lead coating and others without. The lead coating was undoubtedly a good lubricant for automatic coiling, but lead coated springs would be unacceptable in the food trade. It was stated that steel spring wire invariably had a coating, usually copper or phosphate, to facilitate easy coiling, and it was possible to cure a bundle of wire which would not coil by having it phosphated, when it would coil perfectly. In the main, dry lubricants seemed to suit automatic machines rather better than fluid ones. In the U.S.A. drawing through lime and soap produced a grey finish suitable for automatic coiling, and good results had been reported in certain cases using molybdenum disulphide. The Director intimated that a sub-committee dealing with plant and production had on their agenda the investigation of designs of automatic machines, and this might eventually alter the lubrication requirements of the spring makers.

Spring Steel Wire Specifications

On the subject of British Standard Specifications for spring steel wire, the following reasons were given for their infrequent use: (*a*) the spring users did not quote B.S. Specifications and the spring makers had to order wire as specified by the customer; and (*b*) loopholes revealed since the institution of standards for springs in 1940 had rendered the specifications inadequate. Mr. Haynes said the C.S.F.R.O. were issuing a book containing all the specifications dealing with springs and spring materials, and there would also be one to two relevant foreign specifications included.

In response to a query as to whether patented steel wire would have to be replaced by alloy steel wire for the manufacture of valve springs for internal combustion engines where the tendency to increase operating speeds subjected the springs to much higher temperatures than before, the President said it was not possible to give a definite answer at present. There was some difference of opinion as to whether the limiting factor was the fatigue resistance or the limit of proportionality, with its influence on the natural frequency of the spring. Equipment being installed in the new research laboratories would include facilities for stress relaxation studies and fatigue investigations at temperatures up to 600° C., so that the organisation would be appropriately equipped for research of this nature.

American and Continental practice favoured the use of pre-tempered wire for spring manufacture, in contrast to the hard drawn patented steel wire used here, and the possibility of a change-over to pre-tempered wire was discussed. The continued use of patented steel wire in Britain was thought to be due to the material being superior to the same type of wire produced in the U.S.A. or on the Continent. Moreover, it was pointed out that pre-tempered wire was very susceptible to hydrogen embrittlement, both from plating and from the corrosion of an unprotected spring. The President stated that as members of the Organisation, the spring makers were in a position to negotiate and specify what they required, and there would, therefore, probably be an increasing demand for oil tempered wire.

The Development of Beryllium for Nuclear Reactor Applications

By G. C. Ellis, B.Sc., M.Sc., F.I.M.

Atomic Weapons Research Establishment, Aldermaston

There is a considerable interest at present in the use of beryllium as a canning material in nuclear reactors, for which purpose it has attractive nuclear and high temperature properties. Its use in this field presents formidable development problems due to the difficulties encountered at various stages in the chemical and metallurgical processing of the metal. In the first part of this article the author discussed the demand for beryllium in this field, toxic hazards, and the initial production of the metal from the ore. The present section deals with the production of ingot and powder metal, fabrication, and mechanical properties: the possibility of improving the ductility will be considered in the December issue.

(*Continued from page 176 of the October issue*)

Ingot Production Route

BERYLLIUM ingots are produced by high frequency vacuum induction melting and casting, using a beryllia crucible, a graphite or mild steel mould (fitted with a refractory top) and a bottom pouring technique. Vacuum melting and casting reduces the halide and the volatile metallic contents of pebble and flake beryllium metals. American pebble metal ingot contains 99·5% beryllium, with 0·05% magnesium, 0·11% iron and 0·06% aluminium as the main metallic impurities. The fluoride content is not quoted. The beryllium oxide content is 0·2% (or less). American flake ingot contains 99·8% beryllium, 0·003% magnesium, 0·005% iron, 0·005% aluminium, 0·01% chloride and 0·1–0·2% beryllium oxide.

Beryllium oxide is the most inert crucible material for containing liquid beryllium. It is significant that the oxygen content of contaminated scrap beryllium can be reduced to the normal ingot content (0·2%) by repeated melting in a beryllia crucible. This was reported by the Americans,[4] and has been confirmed by A.W.R.E.

In the U.S.A., the early production development at the Massachusetts Institute of Technology of ingot beryllium for reactor use was confined almost exclusively to pebble metal. This work was relatively unsuccessful, and was abandoned in favour of the Brush Company's commercial production of beryllium powder. The Beryllium Corporation at present satisfies a limited demand for fabricated ingot metal and also produces the powder metal.

Ingot beryllium is very brittle at room temperature, and exhibits a coarse grained columnar structure. Beryllium also exhibits hardness anisotrophy. These qualities cause high tool wear and a relatively poor machined finish, the latter being due to individual grain detachment and to mosaic pattern formation. The main difficulty in ingot production is the elimination of unsoundness. Ingot beryllium is hot short, while liquid beryllium possesses a relatively high surface tension, and a low fluidity; excessive solidification rates thus causes the formation of radial inter-columnar cracks and secondary pipe, due, respectively, to low fluidity and poor feeding. On the other hand, the use of slow solidification rates is precluded by the associated accentuation of grain coarseness. The formation of secondary pipe and inter-columnar cracks can be avoided, however, with reasonable consistency, by the use of intermediate solidification rates. The main unsoundness defect exhibited by ingot beryllium is a hair line crack extending axially from the pipe cavity, which the Americans have, so far, failed to eliminate in ingots of 8 in. diameter, or greater.

It appears, therefore, that ingot metal is not yet suitable for the production of large machined blocks for moderator or reflector use. It would also appear to be a relatively unattractive material for fabrication to sheet and tube components. Nevertheless, there is a definite potential demand for ingot beryllium in view of its higher purity and lower cost compared with the powder metal. Grain refinement, if successful, would be expected to confer improved soundness and machinability on the cast metal, together with improved fabrication characteristics and fabricated properties. So far, no success has been reported by the Americans in grain refinement by alloying metal additions, by mould vibration, or by variation in melting practice.[4]

The complete deoxidation of liquid beryllium has been attempted by additions of titanium and zirconium, but without success.[11]

A wide investigation has been made of beryllium alloys.[11] Beryllium-rich solid solutions, which are few, are of limited solubility, and are terminated usually by intermetallic compound formation. These alloys are brittle in both the cast and the fabricated conditions.

Powder Production Route

In the U.S.A., Q.M.V. beryllium powder is produced commercially by the Brush Beryllium Co., at a capacity of several tons per month. Vacuum cast pebble metal ingots, 9 in. diameter (70 lb. weight) are turned to chips by multiple-cutter lathe operation. The chips are milled to powder in a Braun type attrition mill provided with beryllium-faced water-cooled rotating discs operating in an inert atmosphere of nitrogen. The powder is discharged continuously on to a 200 mesh

screen, the + 200 fraction being recycled to the mill feed. The powder is recycled approximately ten times before complete discharge. Since the main characteristics of a powder governing its consolidation and its consolidated properties are its particle size distribution and chemical composition, powder produced in initial lots is evaluated and then blended (according to specified particle size and chemical composition) into batches for final use. The particle size distribution of the powder is approximately 50% −200 to +325 mesh, and 50% −325 mesh, with an average particle diameter of 25–36 microns; its composition is stated to be based on the purity necessary for nuclear and corrosion resistance requirements. Average Q.M.V. powder contains 0·15% iron and 0·05% each of aluminium, magnesium and silicon. The fluoride content is not quoted. The beryllium oxide content increases from 0·2% in the ingot to 0·3% in the chips, and further increases with each successive cycle in the mill to 0·8–1·0% in the final −200 mesh powder.

Experimental beryllium powders prepared in the U.S.A. by the vapour phase reduction and the electrolytic reduction of beryllium chloride exhibited finer particle size and lower metallic impurity content than the standard Q.M.V. powder.[4] These powders proved unacceptable to the Americans for production purposes, owing to their residual halide content. It is rather surprising that very little attempt appears to have been made to reduce the chloride content of these powders by suitable process control, or by the use of a final purification treatment. The purification of electrolytic flake metal by vacuum melting to ingot prior to powder production has not been reported in the U.S.A.

It is interesting that, as opposed to other reactive metal powders, such as those of titanium, zirconium and uranium, beryllium powder in the normal particle size range does not appear to involve a pyrophoric or explosive hazard.

Beryllium powder is normally consolidated by die pressing according to one of the following methods :—

(1) cold pressing at 60–80 tons/sq. in., followed by sintering in vacuum at 1,125°–1,175° C.

(2) (*a*) hot pressing at 1,100° C. using low loads (< 150 lb. sq. in.) in mild steel containers, with long compensating times (24 hours), which in turn demand the use of vacuum, or

(*b*) hot pressing at higher loads (1–5 tons/sq. in.) in special high temperature alloy dies at slightly reduced temperatures (850°–1,000° C.) for shorter times, thus eliminating the necessity for the use of vacuum.

(3) warm pressing at still higher loads (10–20 tons/sq. in.) at further reduced temperatures, 500°–600° C. (below the recrystallisation temperature) for short times, so as to permit the use of tool steel die materials.

The combination of high pressures and high temperatures required for consolidation, together with the abrasive nature of beryllium powder, impose severe demands on die materials. For this reason, and owing to the lack of demand for small parts prepared to near-finished dimensions, consolidation methods (1), (2*b*) and (3), above, are used only for experimental purposes. Commercial production at the Brush Beryllium Co. is confined to vacuum hot pressing in mild steel containers. Large blocks, 5 × 2 × 0·5 ft., weighing 750 lb., and cylinders up to 28 in. diameter are produced in this manner, from which small parts are machined. The dry machining swarf is recovered for powder production purposes. For the experimental production of tubes of plain and longitudinal finned design, a modification of the vacuum hot pressing technique has been used, in which the powder, contained between thin wall mild steel concentric tubes is first evacuated and then consolidated at 1,000°–1,100° C. by an external gas pressure of 500 lb./sq. in.,

At the Atomic Weapons Research Establishment, Aldermaston, the technique of pressureless-sintering loose beryllium powder (45–50% bulk density) to high densities of 95–98% has been developed and established on a production basis[13]. The method consists in sintering −200 mesh powder of critical particle size distribution (about 5·0 microns average particle diameter) at 1,200°–1,250° C. for six hours in a graphite mould heated by induction in a vacuum furnace. Only superficial reaction occurs with the graphite. Hollow shapes are made by sintering on a graphite core. The powder is produced by milling (in stainless steel mills with steel balls) standard quality swarf prepared by the lathe turning of vacuum cast ingots of electrolytic flake beryllium. A purified argon atmosphere is used for milling, powder handling and mould filling. In view of the high oxidation tendency of beryllium and the relatively high consolidation pressures normally required, it is surprising that the consolidation of loose powder to such high densities is possible.

The A.W.R.E. technique offers marked advantages over the commercial method of vacuum hot pressed powder production. The powder is produced in standard milling equipment with short milling periods (20 min.), and it exhibits higher purity (0·05% iron, 0·001% chloride and 0·3–0·6% beryllium oxide) and higher apparent particle size consistency than the commercial powder. The pressureless sintering method of consolidation offers relative simplicity, economy and versatility of production of a wide range of shapes. No press is required. Relatively cheap and readily machinable graphite is used for moulds, and several of the same (or different) shapes can be sintered at the same time, by the use of a multi-cavity mould. Further economy can be effected by the use of twin furnaces with a single vacuum pumping system and high frequency supply, so that one mould is being cooled and recharged while the other is being heated.

Against these advantages, the pressureless sintering technique compared with the vacuum hot pressing method requires a powder of more critical particle size and allows less control of sintered dimension. Neither of these points is particularly serious, and both are capable of improvement. It should be noted that pressureless vacuum sintering at 1,200°–1,220° C. for six hours results in a fine grained metal.

The A.W.R.E. product is well suited for reactor applications in the sintered and machined condition, or after fabrication to tube or sheet (with consequent improvement of mechanical properties). Tubes, bars and blocks can be sintered for canning, structural component and moderator applications and sintered cylinders and slabs can be extruded to tube and rolled to sheet. Considerable importance is attached to the possible preparation of fuel elements by the direct sintering of loose powder around a uranium ceramic fuel core. This has been achieved experimentally on small simulated fuel elements, but certain difficulties remain to be solved for

large scale application. A major attraction of the process is that it eliminates the necessity of joining, as is required with the use of extruded tube.

In comparison with ingot beryllium, vacuum hot pressed or pressureless sintered powder offers distinct advantages for reactor use. It permits the consistent production of sound parts, with little restriction on maximum size. The powder products exhibit a fine grained structure of random orientation, which leads to good machinability and surface finish, together with relatively good fabrication characteristics and fabricated properties.

Fine grain size is the distinguishing feature of consolidated powder. It is attributed to the prevention of grain growth by the presence of inter-granular oxide films during consolidation at high temperatures. For a given consolidation temperature, the ultimate grain size is directly related to the original powder particle size. The 200 mesh particle size used by the Brush Co. is stated to represent a compromise between the better tensile properties obtained at room and slightly elevated temperatures with fine grained metal and the better properties at high temperatures obtained with larger grain size.

In the U.S.S.R.,[3] reactor moderators appear to be produced by the vacuum hot pressing of powder in graphite dies at 50 kg./sq. cm. at 1,100° C. The powder is prepared from electrolytic flake ingot metal.

Cost Data

Beryllium is an expensive metal, even when considered on a volume basis. The following cost data quoted by one firm for <100 lb. quantities of metal indicate that the main cost of production up to the ingot or powder stage arises in the halide treatment of the oxide.

Beryllium oxide	£3 £5 per lb.
Pebble metal	£24 per lb.
Ingot metal	£25 per lb.
Powder metal	£30 per lb.

For comparison purposes, the price of electrolytic flake beryllium produced by the Pechiney Co. is in the region of £36 per lb. A reduction in cost would be expected from an increase in the volume of production.

Fabrication to Tube and Sheet

The two major problems associated with the fabrication of beryllium to good quality tube and sheet are :—

(1) compliance with specified form and tolerances, and

(2) control of grain orientation to reduce property anisotropy with the object of improving transverse properties.

Beryllium in the form of ingot, loose powder or consolidated powder may be used for fabrication purposes, although the use of loose powder is not recommended owing to the greater potential toxic hazard involved. There are certain problems common to both ingot and consolidated powder in their fabrication to the desired form and tolerances. The relatively low ductility and cohesive strength of beryllium at high temperatures result in a tendency to fracture, particularly when deformed in tension. The greatest success is thus achieved with extrusion, which provides lateral support during fabrication and allows deformation mainly in compression. The tendency of beryllium to die tool seizure, and the onset of the toxic vapour hazard and of rapid oxidation at about 800° C. are overcome by the complete containment of the metal in mild steel welded sheaths. The possibility of reaction with the sheath material limits fabrication temperatures to a maximum of 1,075° C. Cold finishing operations for improved surface and tolerances are precluded in both sheet and tube fabrication by inadequate ductility. This limitation is partly overcome by warm working at 400° C., in the region of the first ductility peak, i.e., below the recrystallisation temperature (~ 700° C.). In ingot fabrication, defects also occur due to the non-healing of solidification cracks and to the initiation of cracks at inter-columnar zones of weakness. A greater ease of deformation would be expected with fine grained powder beryllium, due to more uniform stress distribution. The densification which occurs on the fabrication of loose powder causes sheath-folding difficulties.

The forging of ingots for grain refinement prior to extrusion or rolling is not normally possible due to failure by cracking. Bars, of round and rectangular section, and tubes are produced by the sheath extrusion of ingot and consolidated powder beryllium at 1,050° C., with reductions of the order of 30 : 1 ; the extruded surface of the ingot is rough in comparison with that of the extruded powder. Flake metal and machining swarf may be completely consolidated by hot sheath extrusion. Warm extrusion around 450° C. requires the use of a suitable sheath or a surface coating which will act as a barrier and lubricant. Some success has been achieved in bare extrusion at 450° C. by the use of slow speeds and special die materials, but there has been no attempt to dispense with the sheath in hot extrusion by the use of glass lubrication according to the Ugine Sejournet process. Sheet is produced from ingot or powder billet but hot sheath rolling at 900°–950° C., with 10–15% reductions and inter-pass reheating. After a total hot reduction of 75–80%, finish rolling can be carried out at temperatures down to 450° C. Limited reductions by cold rolling are also possible. Tube extrusion appears to give greater consistency of production than does sheet rolling ; powder billet is preferable to the ingot metal for the same reason.

The greatest problem in the fabrication of beryllium ingot and powder billet arises from the necessity for the improvement of mechanical properties (particularly ductility), in the direction transverse to that of working. This is discussed more fully in a subsequent section. Beryllium develops maximum properties in the direction of working. The limited modes of deformation available result in marked property anisotropy, with relatively poor transverse strength and ductility. The attainment of maximum properties in two directions is, therefore, dependent on the development by fabrication control of a suitably orientated structure. This may be obtained in sheet production by the alternate cross rolling of ingot or powder billet, or by the cross rolling of an extruded flat section. The realisation of the same degree of orientation control in tube production is not possible owing to the general tendency to a uni-directional mode of deformation in tube forming processes. Nevertheless, a small but acceptable improvement of transverse properties should be possible, as there is a limited element of transverse working in most tube forming processes. A further difficulty lies in the determination of the transverse properties of the tube.

Joining

Beryllium is a difficult metal to weld or braze because of its cold brittleness, hot shortness, and the ease with

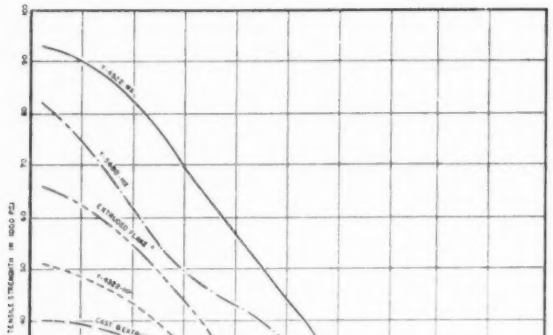


Fig. 2. Comparison of elevated temperature tensile strengths exhibited by various types of beryllium.[15]

which it oxidises. In the U.S.A. fusion welding has not been successful, mainly because of crack formation caused by hot shortness. The best results are reported to be obtained with a bare beryllium filler rod, a tungsten electrode, an inert gas shield and a D.C. current.[4] Pressure welding at high pressures and high temperatures is reported to provide a sound joint of good strength,[4] although the ductility of the joint is not stated. The main difficulty is the provision of suitable jigs, which tends to preclude the pressure welding of awkward shapes. Another difficulty is that the high pressures and temperatures required for a successful joint are not compatible with the joining of thin sections of intricate design.

The Americans consider that brazing is the most satisfactory method of joining beryllium to itself and to other metals.[4] The best results are quoted for a 12% silicon-aluminium alloy electrode, used with an intert gas shield. The furnace brazing of simple shapes is satisfactory using aluminium, silver and aluminium-silver and silver-copper alloys. The elimination of porosity is the main problem here. A notable recent development which may have applications to beryllium is the fusion welding of various high temperature metals in thin sections by electron bombardment in high vacuum.[14]

Unless a method such as the pressureless sintering of powder can be used for the complete sheathing of reactor fuel, the present lack of a reliable joining method presents a formidable problem in the development of beryllium for canning applications. The use of a brazing method is not attractive because of the inevitable increase in neutron absorption and the deterioration in high temperature resistance of the joint. The pressure welding of thin sections is limited by the possible requirement of high deformations. The major problem in the joining of beryllium tube and sheet is the production of a sound gas-tight joint without the loss of orientation-dependent ductility and a reversion to the brittle cast structure of the metal.

Machining

Beryllium is abrasive and causes relatively high tool wear.[4] The metal can be turned, milled, drilled, reamed and ground under conditions somewhat similar to those for cast iron, although it is particularly difficult to saw. Dry cutting conditions, with carbide-tipped tools are preferred for improved tool life, and machined surface finish.

Provided adequate care is taken, the cast metal can be machined to a reasonably good surface, despite a tendency for the detachment of individual grains. The Americans claim that the machinability of the fabricated metal is dependent on grain orientation. The best machinability is exhibited by consolidated powder. It is of interest that pressureless sintered beryllium, when turned under certain conditions, produces long machined turnings, whereas the other forms of beryllium produce only chips on machining.

For the machining of large volumes of metal, economy demands dry cutting conditions for the recovery of the

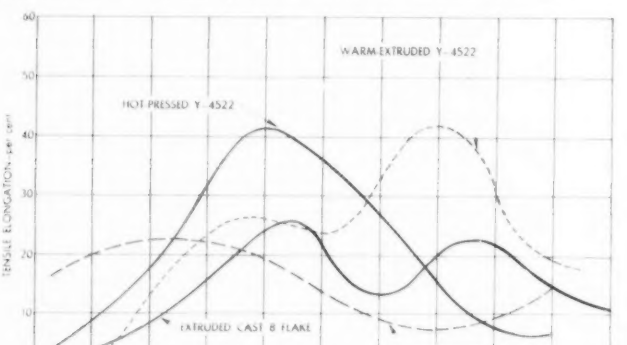


Fig. 3. Comparison of elevated temperature tensile elongation exhibited by various types of beryllium.

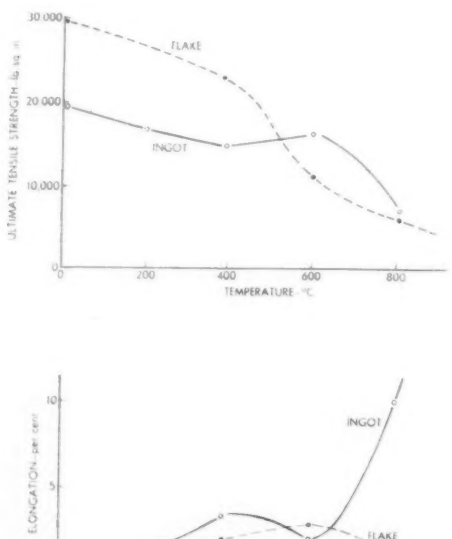


Fig. 4. Transverse strength and elongation of extruded ingot and flake.

clean chips without devaluation for powder production. This is achieved at A.W.R.E. by carrying out the machining within a protective box, with the complete removal of the swarf by a high speed local extract (~ 3,000 ft./min.) at the cutting tool, and the recovery of the swarf in individual cyclones. The extract is sufficient to maintain a minimum face velocity of 200 ft./min., through the hand apertures in the box.

Corrosion Resistance

Beryllium does not oxidise appreciably below 600° C., although fairly rapid oxidation commences around 800°–850° C. In the U.S.S.R., protective coatings are being developed for high temperature use.[3] Atmospheric corrosion at room temperature appears to be connected with the hydrolysis of carbide and halide inclusions in the metal. The water corrosion resistance of beryllium appears to be very erratic, although that of the powder metal seems to be better than that of the ingot. The U.S.A.E.C. acceptance test for Brush Q.M.V. powder blocks is 96 hours exposure at 250° C. There are no data published in the U.S.A. or the U.K. on the corrosion of beryllium by carbon dioxide.

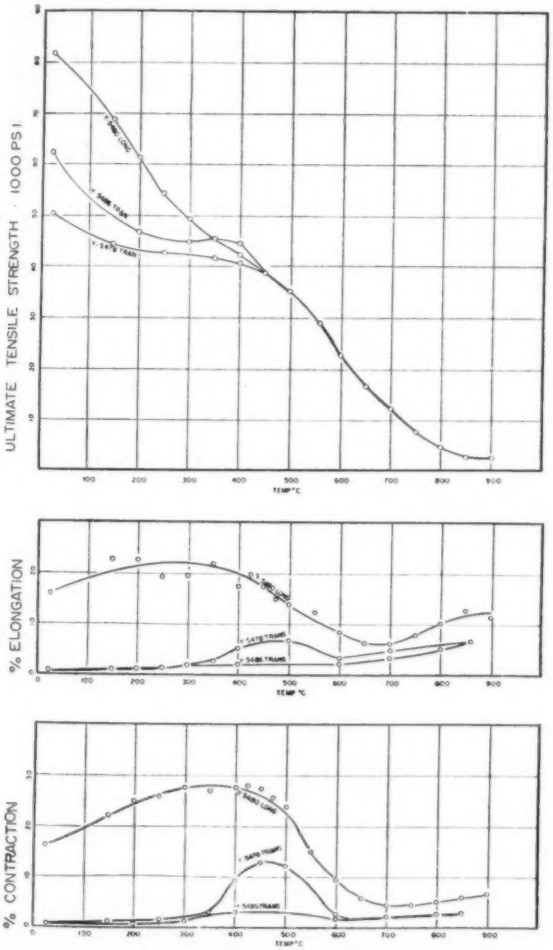


Fig. 5.—Comparison of longitudinal and transverse tensile properties of hot extruded QMV beryllium at elevated temperatures.[15]

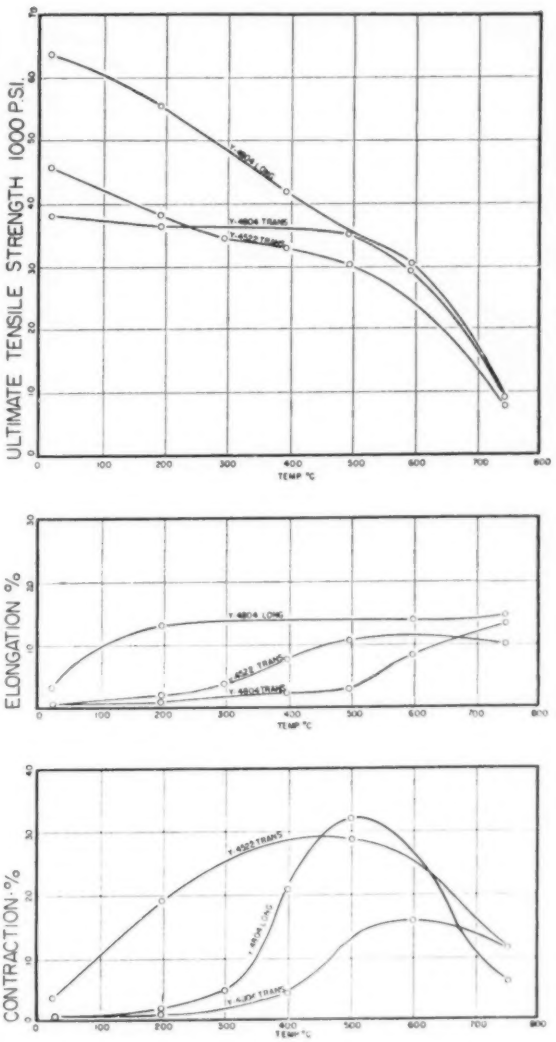


Fig. 6. Comparison of longitudinal and transverse tensile properties of extruded QMV beryllium at elevated temperatures. Vacuum hot pressed and extruded from 2¼ to 1½ in. diameter at 425 C.[15]

Mechanical Properties

At room temperature, ingot beryllium is very brittle, while consolidated powder exhibits only 2–3% elongation. Better ductility values (5–7% elongation) are obtained with the A.W.R.E. pressureless sintered powder. The best properties are developed in ingot and powder by fabrication. The usefulness, from the reactor viewpoint, of both types of fabricated products is limited, however, by poor transverse ductility, especially in fabricated ingot. A further limitation of fabricated ingot is its relatively poor longitudinal ductility, while with fabricated powder, the variability of its moderate longitudinal ductility values causes concern. Thus, extruded ingot exhibits 2–3% longitudinal elongation, with zero transverse ductility, and extruded powder exhibits 5–20% longitudinal elongation, with 2–3% transverse elongation.

For reactor canning and structural component applica-

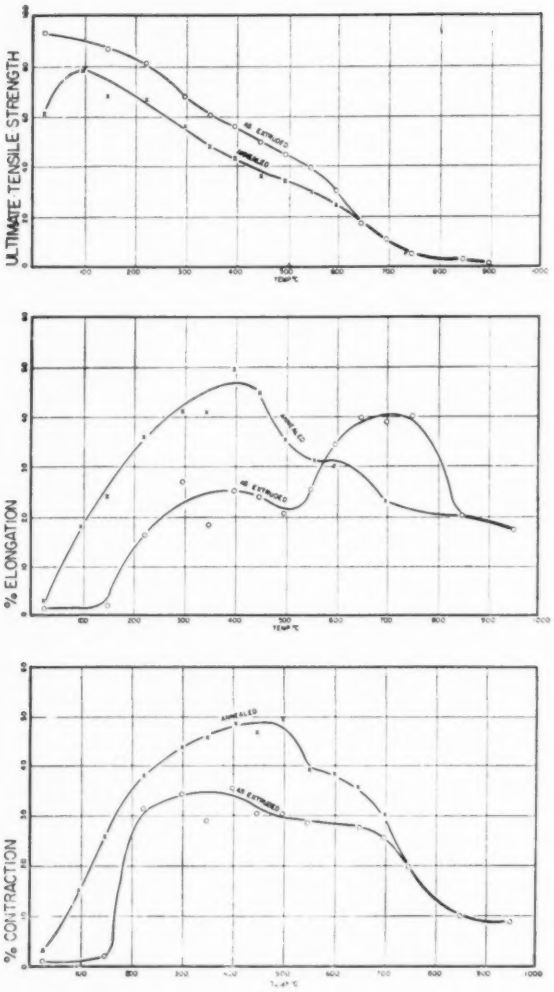


Fig. 7. Elevated tensile properties of vacuum hot pressed QMV beryllium extruded 1 to ½ in. diameter at 425 C. as extruded and annealed at 750 C., lot Y 4522.[15]

tions, an important property requirement is good longitudinal and transverse strength and ductility at all temperatures within the reactor range. Unfortunately, the available American hot tensile data are limited to those of extruded ingot and powder rod. No data are available on the properties of tube or sheet, or of beryllium of higher purity than the commercial grade metal. The available data relate almost exclusively to beryllium of pebble metal origin ; few properties are quoted for the higher purity commercial flake metal. Even so, the following illustrations (reproduced from U.S. publications) serve to indicate the degree of property anisotropy of extruded beryllium, and its variation with increase of temperature.

Fig. 2 gives a comparison of the tensile strength in the longitudinal direction of various forms of fabricated beryllium as a function of temperature.[4] Hot extruded powder (HX curve) is stronger than hot pressed powder (HP curve), which is stronger than extruded ingot. Warm extrusion (WX curve) below the recrystallisation temperature results in a marked increase in tensile strength.

Fig. 3 compares the elevated temperature tensile elongation in the longitudinal direction of various fabricated forms of beryllium.[4] Certain features are of particular interest. These are the sharp increase in elongation with initial temperature rise to 400° C., and the twin-peak ductility curve which is exhibited by all forms of extruded beryllium, but not by the hot pressed powder. It is also of interest that above 350° C. hot extruded ingot exhibits higher elongation than hot extruded powder (Y—5,480). This variation in ductility throughout the reactor range of temperature is somewhat disturbing.

Fig. 4 gives the transverse tensile strength and ductility of hot extruded ingot and flake beryllium up to 800° C. The low transverse elongation ($< 5\%$) up to 600° C. should be noted. A comparison of Figs. 3 and 4 shows that increase in temperature causes a tendency towards isotropy of tensile strength, but leads to anisotropy of ductility.

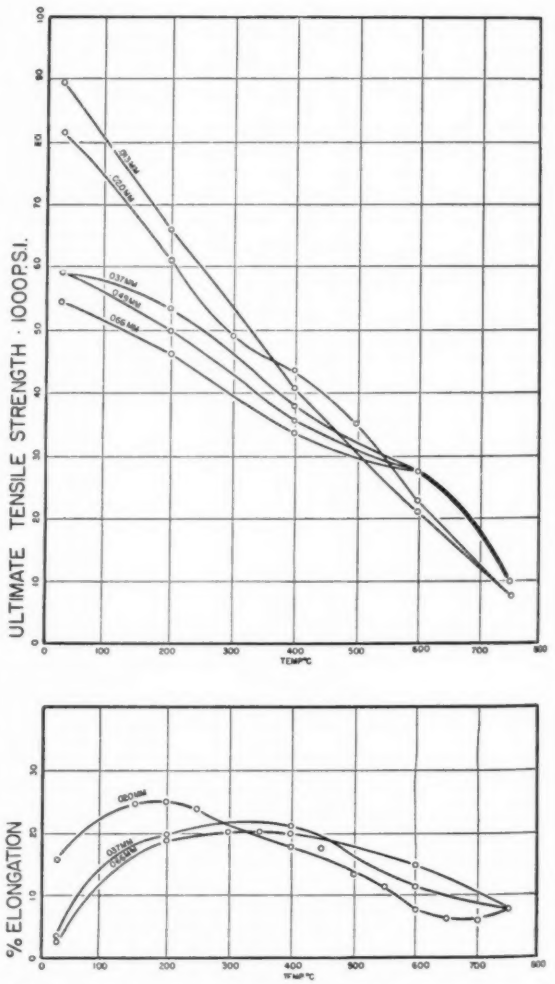


Fig. 8. Effect of grain size on elevated temperature tensile properties of hot extruded QMV beryllium. Grain size in average diameter.[15]

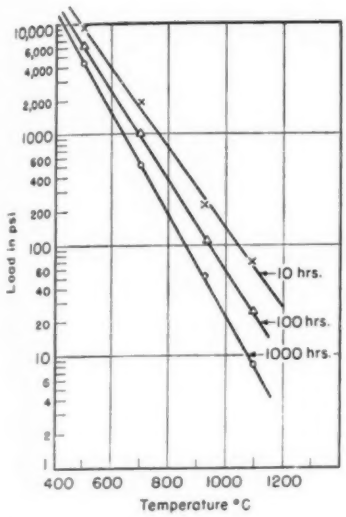


Fig. 9.—Beryllium stress versus temperature curves for arbitrary rupture times.[11]

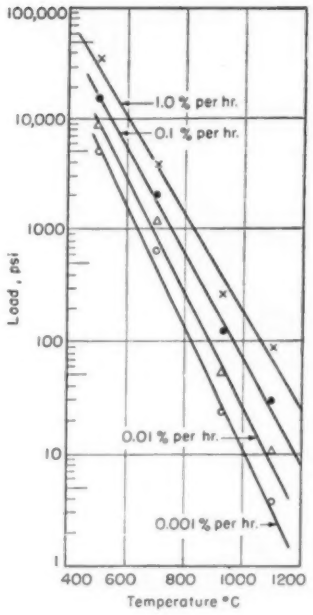


Fig. 10.—Beryllium stress versus temperature curves for arbitrary creep rates.[11]

Fig. 5 shows the longitudinal and transverse tensile strength and elongation of hot extruded powder as a function of temperature.[15] As with hot extruded ingot (Fig. 4), isotropy of strength is obtained at 450° C., while the transverse ductility values remain low (2–3% elongation) with increase of temperature.

Fig. 6 gives the effect of temperature on the longitudinal and transverse tensile strength and elongation of vacuum hot pressed powder, warm extruded at 425° C.[15] In correspondence with hot extruded ingot (Figs. 2 and 4) and hot extruded powder (Fig 5), isotropy of tensile strength is obtained at 500° C. Again, the transverse ductility remains low, with a slight increase at 300° C.

Fig. 7 shows the effect of a recrystallisation anneal (at 750° C.) on the longitudinal tensile strength and elongation of vacuum hot pressed powder warm extruded at 425° C. These data are most encouraging: annealing causes a reduction in strength, but a marked increase in the 400° C. peak ductility value from 25% to 47% elongation.

The extent of the scatter in the properties of fabricated beryllium is evidenced by a comparison of the longitudinal elongation values of warm extruded powder given in Figs. 3, 6 and 7. At 400° C. these vary between the limits 14% and 25%.

Fig. 8 shows the effect of grain size on the longitudinal tensile strength and elongation of hot extruded powder.[15] Decrease in grain size (obtained by finer powder particle size) results in superior strength up to 500° C. and superior ductility up to 350° C. Above 450°–500° C., a relatively coarse grained structure exhibits the higher tensile strength and elongation.

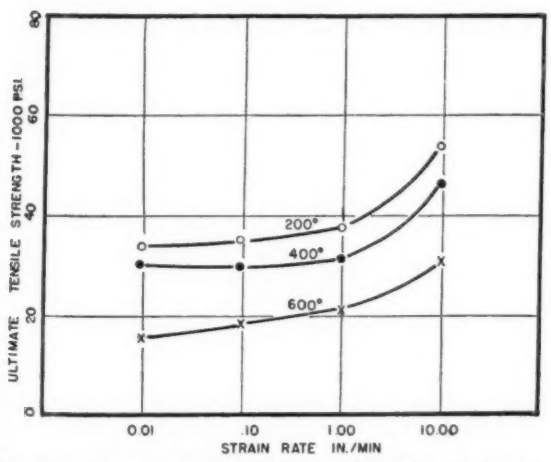


Fig. 11.—Effect of strain rate on the elevated temperature tensile properties of vacuum hot pressed beryllium, lot Y-4522-HP.[15]

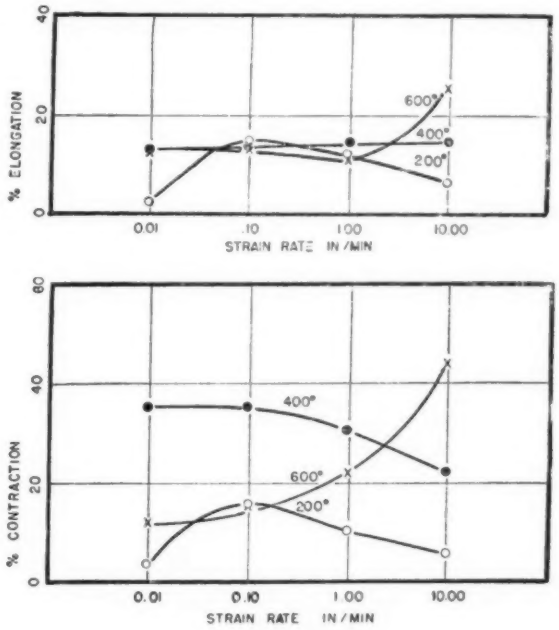


Fig. 11.

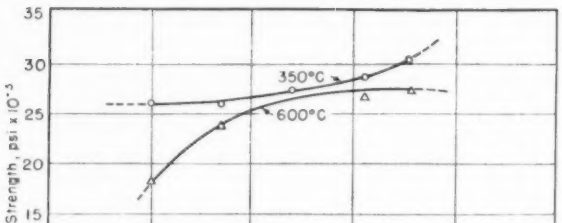


Fig. 12.—Effect of strain rate on ultimate strength of beryllium at various temperatures.[11]

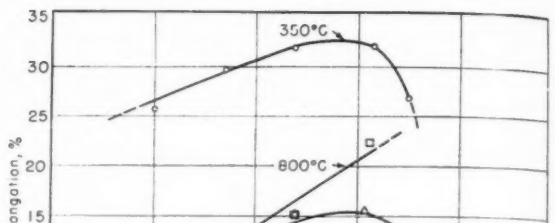


Fig. 13.—Effect of strain rate on elongation of beryllium at various temperatures.[11]

Thus, the American tensile data on extruded beryllium may be described as rather disturbing from the reactor design viewpoint. The longitudinal ductility exhibits a twin peak curve with a minimum value at 600° C., while the transverse ductility of both extruded ingot and extruded powder remains low (<5%) up to 600° C. At 400°–600° C., hot extruded ingot exhibits higher longitudinal ductility than hot extruded powder, but lower ductility than warm extruded powder. Warm extruded properties, particularly after a recrystallisation anneal, are very interesting. It is unfortunate that comparative tensile data on warm extruded ingot beryllium are not available.

As might be expected, beryllium is markedly notch sensitive. Careful specimen preparation and axial alignment in testing are necessary, therefore, to avoid erroneous results.

The published stress rupture and creep data on beryllium are restricted to those of Kaufmann, 1950.[11] These relate to hot extruded pebble metal ingot tested at 400°–1,100° C. The results are reproduced in Figs. 9 and 10. At 600° C. the stress for rupture at 100 hours and 1,000 hours is approximately 2,500 lb./sq. in. and 1,500 lb./sq. in. respectively. There are no corresponding data published on flake ingot or on powder in the extruded or rolled conditions. It is of interest, however, that a decrease in rate of strain at temperatures of 200°–600° C. causes a reduction in tensile strength and elongation with hot pressed powder[15] (Fig 11). A similar effect, Figs. 12 and 13, was observed by Kaufmann for extruded pebble metal ingot.[11] Particular importance is attached to the effect on the ductility of extruded ingot and powder of further reduction in strain rate below the quoted minimum of 0·01 in./min.

Other unusual properties of beryllium are its high Young's modulus (~40 × 10^6 lb./sq. in.), and low Poisson's ratio (0·1–0·03).

REFERENCES

1 *Chemical Engineering Progress*, 1956, **52**, (10), 397–438.
2 Griffiths, R. F. *Metal Progress*, 1954, **65**, 81–86.
3 Meyerson, G. A. International Conference on the Peaceful Uses of Atomic Energy. A/Conf.8P/633. U.S.S.R. 30th June, 1955. "Technology of Manufacturing Items of Pure Beryllium and Beryllium Oxide for Use in Nuclear Reactors". Translated through courtesy of the U.S.S.R. Government.
4 "The Metal Beryllium". Editors: D. W. White and J. E. Burke. A.S.M., Cleveland, Ohio, 1955.
5 Breslin, A. J. "Control of Beryllium Oxide in the Ceramic Industry". *American Ceramic Society Bulletin*, 1951, **30**, (11), 395–398.
6 Wood, G. B., and Brenner, A. "Electrodeposition of Beryllium and Beryllium Alloys". *J. Electrochem. Soc.*, 1957, Jan., **104**, 29.
7 Bryant, P. S. "Beryllium Production at Milford Haven". Paper on "Extraction and Refining of the Rarer Metals". London, 1957. Institution of Mining and Metallurgy. pp. 319–322.
8 Derham, L. J., and Temple, D. A. "The Preparation of Beryllium Metal by Thermal Reduction of the Fluoride", *ibid.*, pp. 323–336.
9 Kaufman, A.R., and Kjelgren, B. R. J. International Conference on the Peaceful Uses of Atomic Energy. A/Conf.8P/820. U.S.A. 28th June, 1955. "Status of Beryllium Technology in the U.S.A.".
10 Williams, J. "The Powder Metallurgy of Beryllium". Paper in "Progress in Nuclear Energy", 1 (V), Pergamon Press, 1956, pp. 300–304.
11 Kaufman, A. R., Gordon, P., and Lillie, D. W. "The Metallurgy of Beryllium," *Trans. A.S.M.*, 1950, **42**, 785–844.
12 Raynor, G. V. "Beryllium, Beryllium Alloys and the Theoretical Principles Affecting Alloy Formation with Beryllium," *J. Roy. Aero. Soc.*, 1946, **50**, 390–415.
13 Barrett, T. R., Ellis, G. C., Knight, R. A. "The Pressureless Sintering of Loose Beryllium Powder." Powder Metallurgy Conference, London, March, 1958.
14 Stohr, J. A. Fuel Element Conference. Paris, Nov. 1957.
15 Beaver, W. W., and Wikle, K. G. "Mechanical Properties of Beryllium Fabricated by Powder Metallurgy," *Journal of Metals*, 1954, **6**, 559–573.
16 Lee, H. T., and Brick, R. M. "Deformation of Beryllium Single Crystals at 25–500° C.," *Trans. A.S.M.*, 1956, **48**, 1003–1037.
17 Sloman, H. A. "Researches on Beryllium," *J. Inst. Metals*, 1932, **XLIX** (2), 365–390.
18 Pugh, S. F. A.E.R.E. M/R 1290. 1953.
19 Wilman Scott V. To be published soon in "Nature."
20 Kopelman, B., and Bender, H. "The Dissociation of Beryllium Iodide in Platinum Containers," *J. Electrochem. Soc.*, 1951, **98**, 89–93.
21 Cook, M. "The New Metal Titanium," *J. Inst. Metals*, 1953, **82**, 93–106.
22 Treco, R. M. "Some Properties of High Purity Zirconium and Dilute Alloys with Oxygen," *Trans. A.S.M.* 1953, **45**, 872–892.
23 Pratt, J. N., Bratina, W. J., and Chalmers, B. "Internal Friction in Titanium and Titanium-Oxygen Alloys," *Acta Metallurgica*, 1954, **2**, 203–208.
24 Rosi, F. D., Dube, C. A., and Alexander, B. H. "Mechanism of Plastic Flow in Titanium—Determination of Slip and Twinning Elements," *Journal of Metals*, 1955, **5**, 257–265.
25 Churchman, A. T. "The Slip Modes of Titanium and the Effect of Purity on their Occurrence during Tensile Deformation of Single Crystals," *Proc. Royal Soc.*, 1954, **A226**, 216–226.

New Melting Furnace Company

BIRLEC, LTD., and Efco, Ltd., announce that negotiations between them for the formation of a new company, to take over certain sections of their business, have reached an advanced stage. It is intended that the new company shall be called Birlec-Efco (Melting), Ltd., and that its offices shall be near Birmingham, at Aldridge, Staffs.

The intended scope of the company is the design and supply of all types of electric melting furnaces for the ferrous and non-ferrous metals industries, together with smelting furnaces and induction heating equipment. The personnel of the company will be drawn from the appropriate divisions of Birlec, Ltd., and Efco, Ltd., thus combining in one organisation the specialist techniques and extensive experience of both companies, who will continue independently to manufacture their respective heat treatment furnaces and other plant.

Change of Telephone Number

THE telephone number of Wild-Barfield Electric Furnaces, Ltd., has been changed to Watford 26091.

NEWS AND ANNOUNCEMENTS

Waverley Essay Competition Awards

THE SCIENTIFIC ADVISORY BOARD of the journal *Research* have awarded the following prizes for the best entries in the Waverley Gold Medal Essay Competition. The essays had to be based on some recent scientific research or new development giving an outline of the scientific background, the experimental results and its potential application in industry. The choice of subject was left entirely to the author.

The Waverley Gold Medal and First Prize : MR. A. G. C. HALL, of the University of Sydney, and MR. J. G. HAYES of the Royal Melbourne Technical College for " Coloured Patterns in Glass ".

The Second Prize : MR. T. R. MANLEY, of A. Reyrolle & Co., Ltd., Newcastle-upon-Tyne, for " The Irradiation of Plastics ".

Special Prize for an Author under Thirty : MR. M. D. LILLEY, of Alpin & Barrett, Ltd., Yeovil, for " Food Preservation with the Antibiotic Nisin ".

Highly Commended : Mr. G. HEYWOOD, of Norwood Technical College, London, S.E.27, for " Radio Astronomy — an Aid to Education ".

The prizes will be presented to the winners on Monday, December 15th, at 6 p.m. in the Lecture Hall of the Royal Society of Arts, John Adam Street, London, W.C.2.

Powder Metallurgy Group

THE next meeting of the Powder Metallurgy Joint Group of The Iron and Steel Institute and The Institute of Metals will be held at Church House, Great Smith Street, London, S.W.1, on Tuesday and Wednesday, 16th and 17th December, 1958, when there will be a symposium on " The Powder Metallurgy of Ceramic-Metal Materials ". The meeting will start at 6.30 p.m. on Tuesday, 16th December, with an introductory paper by Dr. P. Murray, of the Atomic Energy Research Establishment, Harwell. On the following morning, at 9.30 a.m., the main part of the Symposium will begin and will continue until about 5 p.m. A series of six papers describing original research work will be presented and discussed. Visitors will be welcome and tickets will not be required.

Papers for this meeting will be published in advance, together with the papers contributed to the first two meetings of the Joint Group and summaries of the discussions on them, in a new publication of the Joint Group which will be on sale by subscription in November. Normally there will be two issues of this publication each year, but for 1958 there will be a double number (1958, Parts 1/2). The annual subscription price, post free, to non-members of the Institutes is 25s., and to members of either The Iron and Steel Institute or The Institute of Metals, 10s. per annum. Orders for this publication should be addressed to The Secretary, Powder Metallurgy Joint Group, 17 Belgrave Square, London, S.W.1.

Subscription Tax Relief

UNDER Section 16 of the Finance Act 1958, the membership subscriptions of certain Societies approved by the Inland Revenue are deductable from the emoluments of any employment to be assessed for tax if deducted out of those emoluments and the activities of the Society are relevant to the employment. The Institute of Metals has now been approved by the Inland Revenue as a Society coming within the terms of this Section of the Act. The effect of this decision is that members of the Institute of Metals may claim tax relief on their annual subscriptions provided that the activities of the Institute are relevant to their employment. The Institute's new financial year began on the 1st September and membership elections take place frequently during the year. Particulars of membership of the Institute and forms of application may be obtained from The Secretary at 17 Belgrave Square, London, S.W.1.

1959 Corrosion Show

BRITAIN'S biggest-ever display of anti-corrosion methods and materials will be staged in London next April. It will be the 1959 Corrosion Exhibition in the Royal Horticultural Society's New Hall, Westminster. For four days from 27th April, some seventy exhibitors will show and demonstrate all that is new in the battle against corrosion, the menace which presents Britain with a £600 m. bill each year. The first Corrosion Exhibition was held in October 1957. Encouraged by the success of this three-day show—which attracted 8,000 visitors—the Organisers, the technical journal *Corrosion Technology*, planned an even better show. The 1959 Corrosion Exhibition is the result, and nearly 20,000 sq. ft. of floor space will be used to show a complete range of anti-corrosion products.

Control Gear Sales Tour

DURING October a specially fitted van made a tour of the major iron, steel and aluminium works and the area offices of The National Coal Board in South Wales, demonstrating the products of the Control Gear Division of G.W.B. Furnaces, Ltd. The exhibits included examples of certain types of equipment, together with a display of photographic enlargements showing G.W.B. control gear installation of interest to electrical engineers in the South Wales area engaged in the heavier side of industry.

S.G. Iron Licensees' Conference

FOLLOWING similar Conferences, begun in Brussels in 1951 and held successfully in Atlantic City, Paris, Florence, Birmingham and Essen, the 7th International Conference of S.G. Iron Producers took place in Brussels from September 25th–27th, 1958, under the Presidency of Mr. F. Dickinson, Manager of the Development and Research Department of Mond Nickel Company, Ltd.

Delegates to the Conference numbered 175, representing 13 European countries, America and Africa. Six technical sessions were held in the Salle d'Armes, Palais d'Egmont, and the papers presented covered the following topics : treatment ; founding ; processing ; special S.G. irons ; properties, testing and specifications ; and applications. Technical discussions were followed by showing of the Mond Nickel Company's new film " Spheroidal Graphite Cast Iron."

On the evening of September 25th, the Groupement Benelux des Fabricants de Fonte à Graphite Spheroïdal (Benelux Groepering van Spheroïdal Grafiet Gietijzer

Fabrikanten), under the Presidency of M. Richard, welcomed the delegates and ladies at cocktails in the grounds of the Exposition Internationale. On the evening of September 26th, the Mond Nickel Company were hosts at a banquet held at the Hotel Metropole, and the proceedings closed at mid-day on September 27th with cocktails in the Mirror Room, Palais d'Egmont.

Aluminium from the U.S.S.R.

THE Soviet authorities have informed the Board of Trade that Raznoimport—the foreign trade organisation which alone is empowered to sell Soviet aluminium—will not export more than 15,000 tons of unwrought aluminium to the United Kingdom in the next 12 months. At the same time, the Board of Trade announce that they have decided to take no further action on the application by the Aluminum Co. of Canada, Ltd., for an anti-dumping duty under the Customs Duties (Dumping and Subsidies) Act, 1957.

Corrosion Essay Award

THE Education Panel of the Corrosion Group of the Society of Chemical Industry have again decided to award a prize of 25 guineas for an essay on corrosion science by young authors. The competition is open to those who are not more than 27 years old on the closing date, 31st March, 1959. The subject may be any aspect of corrosion of metals and its prevention, and a length of some 4,000 words is suggested, but reasonable latitude is allowable. Further particulars of the competition may be obtained from the Society of Chemical Industry, 14 Belgrave Square, London, S.W.1.

Personal News

MR. H. SMALLWOOD, Chief Accountant of Davy & United Roll Foundry, Ltd., Billingham, Co. Durham, has now also been appointed Secretary of that Company. Before joining Davy & United Roll Foundry, Ltd., in September, 1956, as Chief Accountant, Mr. Smallwood was Group Chief Accountant of the Jardine Group of Companies in Nottingham.

MR. R. W. DEVEREUX, has been appointed Sales Manager of Warwick Production Co., Ltd. Mr. Devereux, the son of the late Col. W. C. Devereux, C.B.E., the founder of the Almin Group, joined Warwick Production in 1957, after service with various other companies of the Group.

MR. C. F. HUEBNER, Buying Controller of The British Oxygen Co., Ltd., has been elected President of the Purchasing Officers Association for the coming year.

MR. R. TURNER, has been appointed a Managing Director of Johnson, Matthey & Co., Ltd. He relinquishes his position of Managing Director of Mallory Metallurgical Products, Ltd., a subsidiary company, but remains a Director.

MR. J. ROYCE, has recently been appointed to the Board of Hedin Ltd., manufacturers of industrial electrical heating equipment.

UNION CARBIDE, LTD., have appointed MR. C. DUNGER, to the position of Area Sales Manager of the Northern England territory. His office will be located at the Wincobank, Sheffield, Branch.

MR. G. J. N. CHERRY, has been appointed a Director of Foundry Services International, Ltd.

MR. E. A. LANGHAM, The British Aluminium Company's Resident Representative in Australia will be returning to the United Kingdom early in 1959; he will be succeeded by MR. G. A. DANIELS. MR. J. W. G. EMERY, at present Malayan Area Representative, will become Pacific Area Representative on the return of Mr Langham.

ENGLISH STEEL CORPORATION, LTD., announce the following appointments to the Boards of their operating companies: English Steel Forge & Engineering Corporation, Ltd., MR. W. H. STRATFORD, Superintendent, Steel Melting, and MR. R. W. BROCKLEHURST, Superintendent, Heavy Forge Production: English Steel Rolling Mills Corporation, Ltd., MR. P. TURRELL, who will also hold the office of Assistant Managing Director; English Steel Castings Corporation, Ltd., MR. L. R. EVANS, Superintendent, Foundries; English Steel Spring Corporation, Ltd., MR. R. FIELDING, Superintendent, Spring Production.

GEORGE KENT, LTD., announce the appointment to the Board of their Belgian Subsidiary Company, Kent-Continental S.A., of MR. F. J. HAVENITH, as local Resident Director. Mr. Havenith has been the Manager, first of Kent's Brussels Office, and subsequently of the Belgian company, since its formation.

DR. HAROLD MOORE, C.B.E., has been elected an Honorary Member of the Iron and Steel Institute, of which he has been a member for 57 years. Dr. Moore joined the Research Department, Woolwich, as Chief Metallurgist in 1904, and during the ensuing quarter of a century he saw his department grow into one of the country's leading metallurgical laboratories. In 1932, he became Director of the British Non-Ferrous Metals Research Association, a position he held until his retirement in 1944. From 1934 to 1936, Dr. Moore was President of the Institute of Metals, and he was awarded the Institute's Platinum Medal in 1943. He has been actively associated with several technical and scientific societies, being a Founder Fellow of the Institute of Physics and the first President of the Institution of Metallurgists.

MR. A. P. GAGNEBIN, has been appointed a Vice-President and MR. J. M. WELDON, an Assistant Vice-President of The International Nickel Co., Inc. Mr. Gagnebin has been associated with International Nickel since 1930, and joined Inco's Research Laboratory at Bayonne, New Jersey, in 1932. Mr. Weldon first became associated with the Company in 1927, and has held various positions in the sales, executive and other departments.

Obituary

WE regret to record the death of the following:—

MR. C. C. MASON, of the Cambridge Instrument Co., Ltd., who died suddenly on September 9th, 1958, at the age of 77. Mr. Mason joined the Company in 1910, and was Joint Managing Director from that year until his retirement from executive work in 1941: he remained on the Board until his death.

MR. F. A. SPENCE-BROWN, a Joint Managing Director of Johnson, Matthey & Co., Ltd., who died suddenly on September 29th, 1958.

RECENT DEVELOPMENTS
MATERIALS : PROCESSES : EQUIPMENT

Deep Field Lifting Magnet

CIRCULAR lifting magnets provide a well-established means of transporting iron and steel in a variety of forms. The conventional design has a magnetic strength adequate for normal duties in steel works, foundries, rolling mills etc. Often, however, where space is limited, as in the loading or unloading of railway wagons, or where the load is not too responsive to magnetic induction, as in slag handling, a higher magnetic field is demanded in a given diameter. Rapid Magnetic Machines are now making deep field circular lifting magnets of a much deeper construction than is usual, giving up to 100% greater lift, dependent on the nature of the load. The magnets can be used, with the advantage of increased lift and reduced man-hours, in all applications where hitherto standard or conventional types have been customary. One 46 in. diameter model is operating in the slag industry, lifting up to 3 ton lumps of iron-bearing slag or skull cracker balls at anything up to 9 tons weight.

Rapid Magnetic Machines, Ltd., Lombard Street, Birmingham, 21.

Gas Carburizing Furnace

To meet the need for small scale production and laboratory research in gas carburizing prior to large scale production runs, Royce Electric Furnaces, Ltd., have developed a small electrically heated gas carburizing furnace having a work space of 10 in. diameter × 12 in. high. It is nominally rated at 18 kW., and is suitable for a maximum temperature of 1,050° C. Although designed for heavy industrial use in a production line, the unit is also suitable for a laboratory or research establishment. Heating elements of 80–20 nickel-chromium in coil form are housed in open groove tiles in the sidewalls surrounding a gas-tight heat resisting steel retort giving an even heat transference. An automatically cooled fan motor, fitted with a specially detachable spindle, is housed in the base and drives a heat resisting steel fan unit fitted within the base of the retort, which gives a rapid circulation of the carburizing gas and ensures both uniform case depth and temperature distribution. For simple and rapid operation, the plug lid is fitted with a hand-operated power jack and swivel mast : when in the closed position, a sealing lip engages in an annular seal in the gas-tight retort. Cooling vents are provided to give accelerated cooling of the furnace when required.

Carburizing may be carried out by means of a carburizing gas, with or without an enriching gas (propane), or by the thermal decomposition of hydrocarbon fluid injected directly into the work chamber. It is claimed that gas carburizing has a number of advantages over other methods, as the overall heat treatment cycle can be reduced and the storage space necessary for box or salt bath carburizing is eliminated. Further, all parts of the load are brought to carburizing temperature before the gas is allowed to come into contact with the work, giving uniformity of case depth throughout the charge. The equipment is supplied complete with fully automatic temperature control with an indicating controller instrument and hydro-carbon injector unit, or, where consumption demands, a complete enriched gas generator.

Royce Electric Furnaces, Ltd., Sir Richard's Bridge, Walton-on-Thames, Surrey.

Crane-Approach Warning System

IN factories and steelworks where two or more overhead travelling cranes are operating on the same track there is constant danger of collision, because the crane driver must concentrate on his load and the signals of his slinger rather than on the movement of neighbouring cranes. To eliminate this danger and so promote greater safety in industry, a new electronic equipment has been developed (under the auspices of the British Iron and Steel Federation) by B.T.H., and an installation has been undergoing extensive trials at the Redbourn, Scunthorpe, iron and steel works of Richard Thomas and Baldwins, Ltd. The equipment is the first of its kind to go into service in a British factory and the first to be installed in a European steel works.

The B.T.H. system is based fundamentally on the principle of the echo sounder. An oscillator circuit generates short electrical pulses at regular intervals which are emitted in the form of very high frequency sound (just above the threshold of the human ear) by loudspeaker fitted on the crane. The sound pulses are reflected by a special trihedral reflector fitted on the neighbouring crane and the echo is picked up by a microphone mounted alongside the sound source. The

microphone signal is then amplified and fed to relays which may operate audible alarm or visual indicating circuits in the driver's cab, or may be used to initiate automatic braking of the crane. Automatic warning of approach can thus be given to one or more cranes operating on the same track, or to cranes operating at different levels.

An important feature of the equipment is that the operating system can be made to operate at any desired position on the crane track, merely by placing a trihedral reflector in the path of the sonic beam. Over-running at end stops can thus be avoided and—of greater importance—complete protection can be given to maintenance men working on a disabled crane, or on the crane track, for the reflectors weigh very little and can easily be fitted at suitable positions in the path of the working cranes.

The present equipment can be adjusted to operate up to a distance of 60 ft. from the reflector, but longer distances may be accommodated later. The oscillating frequency is 11 kc. s., and the time taken for a return echo to operate the protective system is 2 milliseconds per ft. Selective circuits are designed to make the alarm system unresponsive to any other signals than those of the genuine echo. The system is designed to operate from any D.C. crane supply, but where cranes operate on A.C. suitable rectifiers may be supplied for conversion.

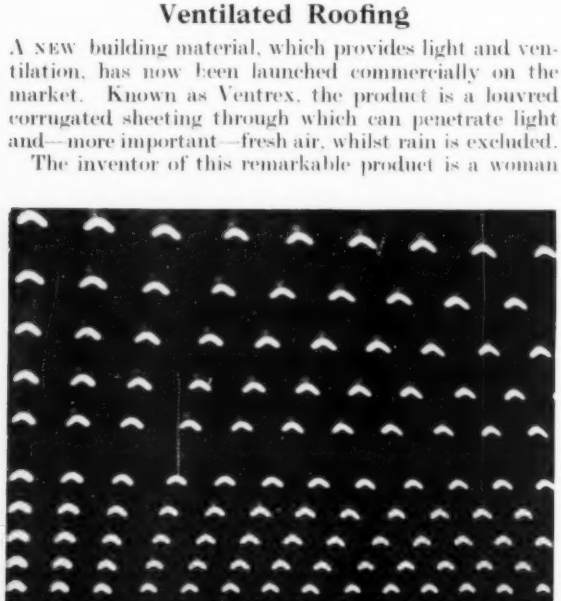
The installation of B.T.H. sonic equipment at Redbourn has proved eminently successful in service, and it has withstood arduous conditions of heat, vibration, and dust without breakdown or reduction in efficiency. It has solved a problem which has been troubling the steel industry for many years, and is an invaluable contribution to industrial safety. Equipment of this type is recommended in the booklet "Crane Safety in the Iron and Steel Industry" recently published by the British Iron and Steel Federation.

The British Thomson-Houston, Co., Ltd., Rugby.

Ventilated Roofing

A NEW building material, which provides light and ventilation, has now been launched commercially on the market. Known as Ventrex, the product is a louvred corrugated sheeting through which can penetrate light and—more important—fresh air, whilst rain is excluded.

The inventor of this remarkable product is a woman farmer, Mrs. I. V. Dawson, of Ringmer, Sussex, who used it on her barns, piggeries and cowshed for a considerable length of time for tests before entering into manufacture. Ventrex is now being made in Galvatite, the John Summers' galvanised steel sheet made by the continuous galvanising process. This method of galvanising, pioneered in this country by John Summers and Sons, Ltd., Shotton, provides a coat of zinc uniform in thickness and ensures the maximum adhesion between the zinc and the steel base, thus giving the longest possible life for the galvanised sheet.



Although light and air seep through the louvres, rain and snow cannot enter even in a high wind. The secret of this remarkable fact lies in the special design of the louvre, which has taken some six years to perfect. Although the use of Ventrex has so far been almost entirely confined to agriculture, factories and stores, its industrial and general potential is extremely wide. Sample quantities are already being shipped to Africa for housing purposes, and its value in industry where noxious fumes are present is being widely recognised.

Ventrex Roofing, Ltd., 5 Fenchurch Street, London, E.C.3.

Automatic Performance Recorder

THE Blick performance recorder was initially developed for use in a manufacturing organization using a form of automation, in which it is electrically connected to a transfer machine, to provide records which can be analysed and used towards increased efficiency and production.

The instrument records the day, the time (to the nearest minute) and the serial number of the component, every time a component passes through the transfer machine. The record is printed on a continuous paper roll which is passing through the recorder at a speed arranged to coincide with the operational performance. A positive "number off" is thus obtained for any given period, thus avoiding any checking necessary if reference is to be made to an operator. In the application for which the instrument was initially developed, however, the transfer machine is left unattended.

The paper roll continues to pass through the recorder as long as the controlling machine is operating, the total time capacity of the paper roll being fourteen days. Should the controlling machine close down at night, however, or cease production for any other reason, either deliberate or accidental, the paper roll will continue to pass through the recorder for three minutes, and it will then stop. When the controlling machine recommences production, the paper roll will start running again in the Blick instrument, recording the first and subsequent operations after the delay. This delayed action device serves a dual purpose. Recording paper is not wasted during shut-downs, and a gap is left in the printed roll, immediately indicating the stoppages to a person inspecting the records. The printed record can be torn from the roll without interfering with the production or recording process.

The Blick recorder can be used as a semi-portable instrument or as a permanent fixture to the controlling machine, or it can be operated by remote control any distance away. A supervising engineer can thus have any number of instruments in his office, from a single recorder to a complete battery, enabling him to check machine performance from time to time, without leaving

his office and without the necessity of interpreting the printed record. Apart from analyses the records are immediately and completely self-evident.

Blick Time Recorders, Ltd., 96–100 Aldersgate Street, London, E.C.1.

Mild Steel Electrode

A NEW iron powder electrode has been introduced for the welding of mild steel. Known as Ferrolux, it is designed for high speed welding in the flat and horizontal-vertical positions. Using a touch-welding technique, the electrode can be drawn out to make long, smooth fillet welds. The fillet shape is mitre with a neat ripple. Slag detachability is good, and the electrode is of value to operators on production work where high output and good weld profile is required.

Iron powder electrodes are claimed to be proving their worth in improving welding productivity by giving increased speeds and longer run lengths when compared with conventional electrodes. To ensure instantaneous arc starting, Ferrolux are lightly tipped at the striking end with a special composition during manufacture (patent applied for). They are fully extruded and are made in 18 in. (457mm.) lengths in sizes from 10 s.w.g. to $\frac{1}{4}$ in. diameter (3·25 to 6·35 mm.). The electrodes can be used with either A.C. or D.C. welding plant, and when the latter is used, the electrode should be connected to the positive pole.

Ferrolux electrodes conform to the requirements of B.S. 639 : 1952 and are approved by Lloyd's Register of Shipping and the Ministry of Transport and Civil Aviation. The following results are typical of those obtained from all-weld-metal test specimens welded with the various sizes of the electrodes in accordance with B.S. 639 : 1952. Yield stress, 29 tons/sq. in. ; ultimate tensile stress, 34 tons/sq. in. ; elongation on 3·54D, 28·5% ; reduction in area, 48% ; Izod impact value (V-notch), 63 ft. lb. ; bend test over 180° without failure.

Quasi-Arc, Ltd., Bilston, Staffs.

Polythene Coated Aluminium Foil

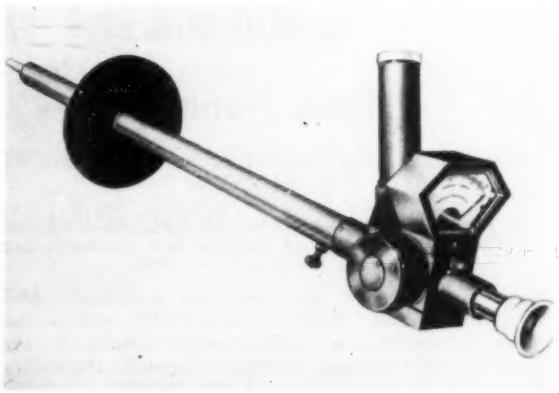
FISHER'S FOILS, LTD., are now making aluminium foil lined with polythene. On to aluminium foil in gauges between 0·00047 and 0·004 in., polythene can be extruded to a thickness of from 0·00075 to about 0·0015 in., supplies being available in reels or sheets up to 30 in. wide. Polythene-coated aluminium foil is an excellent barrier to the passage of moisture vapour and gas, and as it is also heat-sealable it provides a very efficient packaging medium. It is anticipated that many other industrial uses will be found for this new laminate.

Fisher's Foils, Ltd., Wembley, Middlesex.

Molten Metal Temperature Measurement

THE Foster Instrument Company is now marketing a pyrometer known as the Foster-Platt optical immersion pyrometer, which opens up an entirely new approach to the temperature measurement of molten metals in crucibles and ladles. It is claimed that it completely overcomes the limitations imposed on the normal optical pyrometer, and it is extremely simple to operate ; accurate readings can be obtained in a matter of seconds.

The optical axis of a disappearing filament type of optical pyrometer is extended in a light-tight tube sealed at its outer end with a refractory sheath. On immersing



in a bath of molten metal its inner end assumes in a few seconds the temperature of the bath, and since the image of the pyrometer filament is permanently superimposed, a reading can be obtained quickly and accurately regardless of the type of alloy being tested. In most non-ferrous metals the refractory sheath will withstand up to 150 immersions and 50 in cast iron. In the event of the sheath breaking, a new one can quickly be fitted at a small cost. The instrument is completely self-contained, having dry batteries with special high current cells contained in the tubular handle.

Foster Instrument Co., Ltd., Letchworth, Herts.

Barrier Cream Additive

THE increasing use of barrier creams in industry has led to a number of experiments being carried out in an attempt to improve their efficiency, and it has recently been shown that the emulsion type of barrier cream can be improved by the addition of small quantities of P.V.P. (polyvinylpyrrolidone). It is claimed that the P.V.P. has the effect of enhancing the protective qualities of the cream. In addition, it gives a softening action which persists even after the hands have been washed. The addition of P.V.P. substantially assists the removal of dirt or grease and the hands are left in a particularly clean condition.

Experiments which have been carried out in the laboratories of British Oxygen Research and Development, Ltd., at Morden, have shown that if the amount of P.V.P. added to the cream is less than 0·2%, the desired effect is not produced in many cases. On the other hand, if more than 2% is added, the cream has a tendency to make the hands sticky, although the other properties of the cream are not affected. It has been found that the correct amount to use is between 0·5% and 1·5% of the total weight of the cream.

The invention relates only to the emulsion type of barrier cream. This is a cream which is in the form of an aqueous or watery emulsion, the non-aqueous phase of which consists of greasy, fatty or waxy substances. The whole emulsion can be loaded, if required, with an inert filler such as China clay. When the emulsion is applied to the skin, it fills the pores and coats the surface to prevent dirt from entering and also to prevent the skin from being attacked by oils and solvents.

The British Oxygen Co., Ltd., Bridgewater House, Cleveland Row, London, S.W.1.

CURRENT LITERATURE

Book Notice

PRINCIPLES AND PRACTICE OF FLOW METER ENGINEERING

By L. K. Spink. 8th edition, revised and enlarged. pp. 550. Published by the Foxboro Company and obtainable from Foxboro-Yoxall, Ltd., Redhill, Surrey. 108s.

EARLIER editions of 'Principles and Practice of Flow Meter Engineering' have sold some 26,000 copies, so that most engineers will be familiar with the work. In this latest revised and much enlarged 8th edition, the author, the late L. K. Spink, (he died shortly after completing this edition), covers a much broader scope than previously. The book may be said to have reached the status of a text book, though it still retains its usefulness as a handbook for everyday use by practising engineers, particularly those in the oil, chemical and gas industries.

The work is arranged in five parts and deals in great detail with every aspect of flow measurement. Part 1 deals with flow measurement in general, and includes sections on the characteristics of primary devices, the theory of the differential type meter, the installation of primary devices, meter mechanisms, and the choice of head meter specifications. Parts 2, 3 and 4 deal with liquid, steam and gas flow, respectively. Each part follows the same general pattern, discussing the installation of flow meters, methods of calculation and chart computation. Throughout, the text is supported by charts, equations and tables on a generous scale. Part 5 contains a reliable index, a very complete bibliography, a metric units supplement, and an appendix containing lists of working equations grouped for ready reference.

The present edition has been extensively revised. For example, where necessary, symbols have been changed to agree with A.G.A. and A.S.M.E. terminology. In conformity with modern practice, Reynolds numbers corrections have been based on line size, i.e., RD is a function only of flow rate and not of orifice diameter pipe diameter ratio (d D). All tables, factors, etc., in the Natural Gas Section are now based on the A.G.A. Report No. 3—the 'Bible' of the natural gas industry. In addition to these revisions a number of new items appear. These include a discussion of meter functions and mechanisms illustrated by simple line sketches of the various types of measuring, controlling and transmitting mechanisms. There is a very complete presentation of data on radius taps, Dall tubes and eccentric, segmental and quadrant edge orifices. Basic orifice factors for 2–½D and 8D taps, following the A.G.A. Report No. 3 are listed. A new chapter outlines the basic conditions for gas measurement contracts and offers many useful suggestions.

Trade Publications

THE production of accurately cut lengths of tube for subsequent component manufacture, calls for the use of specialist machine tools, and a recent leaflet issued by the T.I. subsidiary, Brookes (Oldbury), Ltd., describes the newest tube cutting off machines which are available in a range of sizes capable of dealing with tubes up to 5 in. outside diameter. A further new Brookes leaflet deals with hydraulic guillotine shears in a full range of capacities from 6 ft. × ¼ in. to 12 ft. × 1 in.

CONSTANT improvements in mill drives give greater production and better quality in the product. Modern drives have such features as individual rectifiers, magnetic amplifier regulators, twin drives with armature loops, and on the tables individual motors on each roll. An interesting article in new concepts in drive systems for hot strip mills, reprinted from *Iron and Steel Engineer*, dealing with these developments, can be obtained from International General Electric Co., of New York, Ltd., Lincoln House, 296-302, High Holborn, London, W.C.1.

WITHIN the last decade or so, chelating agents based on E.D.T.A. have advanced from somewhat limited applications in analytical chemistry to much wider uses in industry, and there is a steadily increasing demand for them for the new uses continually being discovered. A new booklet issued by Whiffen and Sons, Ltd., provides a background to the subject and briefly outlines the properties and uses of the Metaquest E.D.T.A.-based chelating agents made by the company.

THE sixth issue of the *Inco-Mond Magazine* is now available. The leading article deals with the use of nickel-containing stainless steel in the manufacture of fatty acids. Among the other articles are descriptions of a unique air-conditioning process at one of Inco's mines, and the use of iridium type alloys in the manufacture of Swan pens. Other uses of Mond products in portable electric tools, marine chronometers, glass figurines, and equipment used in the dairy for teaching students at Aberystwyth University are featured. *Inco-Mond Magazine* is obtainable free on request from The Mond Nickel Co., Ltd.

Books Received

"Recommended Methods for the Analysis of Trade Effluents." Prepared by a Joint Committee of the Association of British Chemical Manufacturers and the Society for Analytical Chemistry. 124 pp. Cambridge, 1958. W. Heffer and Sons, Ltd. (For the Society for Analytical Chemistry.)

Chambers's Technical Dictionary. Revised Edition with Supplement. Edited by C. F. Tweney and L. E. C. Hughes. 1028 pp. 60,000 terms from 120 branches of scientific and industrial activity. London and Edinburgh, 1958. W. & R. Chambers, Ltd. 35s.

"Introduction à la Physique de l'Etat Solide." By C. Kittel. Translated by E. L. Huguenin and R. Papoular. 594 pp. inc. index and 220 illustrations. Paris, 1958. Dunod. 6 800 F.

"Stahleisen-Wörterbuch." Deutsch-Englisch : Englisch-Deutsch. Foreword by K. Thomas and K. Headlam Morley. 272 pp. Düsseldorf, 1958. Verlag Stahleisen m.b.H. In imitation leather. DM12.50., or 21s. net.: postage 6d., abroad 1s. 4d. Distributing agents for the British Commonwealth and the U.S.A. : H.K. Lewis and Co., Ltd., 136 Gower Street, London, W.C.1.

LABORATORY METHODS
MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC
INSTRUMENTS AND MATERIALS
NOVEMBER, 1958 Vol. LVIII, No. 349

Magnetic Flaw Detection

By A. G. Gardner, A.I.M.

The magnetic particle method of detecting flaws in iron and steel components is now widely used in industry. In this first part of his article, the author discusses briefly the principles on which the technique is based. The second part will deal with techniques and equipment.

MODERN engineering designs make exacting demands on many components, and to ensure satisfactory service life they must be substantially perfect. Fine cracks, whether they originate in the hot working, heat treatment or grinding operations cannot be tolerated, but their presence is often impossible to detect with the naked eye. The necessity for detecting such flaws in ferrous metal parts has led to the development of the magnetic particle method of crack detection, and the importance of this technique has been amply proved in recent years.

The mechanical properties as determined on a sample of material, are no guarantee that a component made from it will behave satisfactorily in service. The component, when designed and manufactured, may contain defects which cannot be discovered by mere visual inspection. Cracks may be present in the surface of a metal which may be so fine that they cannot be seen, even with the assistance of a magnifying glass, and magnetic crack detection offers a means of discovering cracks of this type in magnetic materials such as iron and steel without in any way damaging the component being examined.

Most of the flaws occurring in steel components can be covered by three main groups: (*a*) those arising as a result of forming operations; (*b*) those arising during heat treatment; and (*c*) those arising during grinding. There are other causes of cracking, such as the pickling of hardened steel components, and the welding of unsuitable materials, and once the component has been in service there is a possibility of cracks arising as a result of service strains, whether of a static or dynamic nature.

Forging defects may take the form of surface laps, where the material has flowed under pressure in such a way that the surface has been folded into the body of the metal, thus forming a weakness, which may extend during heat treatment. Again, bursts may be formed at the surface or within the forging as a result of unsuitable working conditions for the material concerned.

When a piece of metal is heated in a furnace, strains are set up due to the temperature of the interior of the workpiece lagging behind that at the surface. Similarly, strains are set up in cooling, and the more rapid the heating or cooling, the more severe the strains induced. In the case of certain metals, of which steel is a notable example, the position is complicated by stresses set up by volume changes accompanying phase changes in the material. The net effect of the generation of stresses in this way may be the formation in the material of heat treatment cracks which may be difficult or impossible to see with the unaided eye.

Efficient grinding depends upon suitable wheel speed, correct pressure, and the right type of wheel grit and bond for the metal concerned. Failure to observe these points results in an inefficient process which is productive of cracks. Grinding cracks are usually the result of excessive local heating by the grinding wheel, and frequently occur in the form of a network.

Basic Magnetic Considerations

Before proceeding to a discussion of the principles involved in magnetic flaw detection, simple definitions of the terms used may be helpful.

Magnets—These have the ability to magnetise and thereby attract iron and other ferromagnetic materials, i.e. materials with magnetic properties similar to those of iron. A simple bar magnet has two poles, one at each end, which are attracted by the North and South Magnetic Poles of the earth. That which is attracted to the earth's North Pole is called the north pole of the magnet and the other the south pole.

There are two types of magnet, permanent magnets and electromagnets. The former may occur naturally as magnetite, but industrially useful permanent magnets are made by magnetising by means of an electromagnetic field suitable ferromagnetic materials. The latter include hardened steels—of the plain carbon type or alloyed with chromium, tungsten, etc.—and special alloys based on aluminium, nickel, cobalt, etc.

Electromagnets usually consist of a ferromagnetic material having about it a winding of one or more turns of an electrical conductor. A material is chosen which has properties quite different from those of a permanent magnet, so that pieces of magnetic material can only be attracted whilst a current flows in the winding. By successive reversals and reduction of the current, an electromagnet can be used to demagnetise a permanent magnet.

Magnetic Materials—The following materials are strongly magnetic: iron and steel (with the exception of austenitic steels), cobalt, nickel, and the permanent magnet alloys. Soft iron and steel (i.e. low carbon) cannot be permanently magnetised to any appreciable extent, and are useful, therefore, in the construction of electromagnets, since they have a high permeability (see below).

Magnetic-Field—The space near the poles of a magnet and that surrounding a conductor carrying current is occupied by a magnetic field. Magnetic fields surrounding the poles of a magnet have polarity, and a magnet having two poles is often called a bipolar magnet, and the entire field a bipolar field. The field surrounding a straight current carrying conductor has no polarity and is called a circular field. When a steel bar is traversed by a longitudinal electric current, the bar is said to be circularly magnetised

Lines of Force—It is convenient to represent a magnetic field diagrammatically by lines of force. These have no real existence, but they provide a useful picture of the properties of a field. Lines of force emanate from the north pole of a magnet and proceed to the south pole, but because of the property attributed to them of repelling each other sideways, they form a pattern of arcs from one pole to the other, some of them apparently leaving the north pole in a direction away from the south pole. The needle of a compass placed in the field will align itself with the lines of force. The flux density of the field is indicated by the number of lines present in a given cross-sectional area.

Magnetic Field Round a Conductor—In the case of a current carrying conductor, the lines of force run in concentric circles round it. The direction of the field is at 90° to that of the current flowing in the wire, and is determined by the corkscrew rule, which states : if a right-hand corkscrew be screwed into the conductor in the direction of the current, the corkscrew turns in the direction of the lines of force.

Solenoid—This is a coil of wire or a succession of loops wound in the form of a helix. When an electric current is passed through the solenoid it acts as an electromagnet and has polarity, its poles acting similarly to those of permanent magnets.

Permeability—This is a term used to express the readiness with which a material can be magnetised, i.e. its magnetising capability. It is numerically equal to the flux density (B) divided by the magnetising force or field strength (H), i.e.

$$\mu = \frac{B}{H}$$

A curve showing the permeability of any material over its magnetising range is called a *B-H* curve.

Saturation—When a specimen being magnetised has reached the point where further increases of magnetising force do not produce any further magnetisation, the object is said to be saturated.

Residual Magnetism—When a specimen is magnetised the flux density induced does not increase uniformly with the magnetising force. The relationship which exists is shown by a typical hysteresis curve, and after the magnetising current has been reduced to zero, the specimen will retain some degree of magnetism called residual magnetism. Retentivity of a specimen is its property of retaining to a greater or lesser degree a certain amount of residual magnetism.

Longitudinal Magnetisation—The simplest example of this is a permanent bar magnet. This has poles at each end, one attracted by the earth's North Pole and the opposite end attracted by the earth's South Pole. The term bi-polar magnetisation is sometimes used in place of longitudinal magnetisation, but the two terms are not really interchangeable : e.g. a magnetised bar may have numerous poles spaced at various distances along its length : these are called consequent poles.

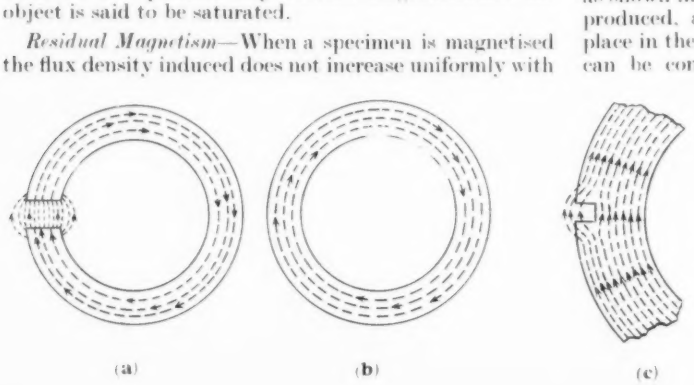
Longitudinal magnetisation is one of the two types of magnetisation used in magnetic crack detection and it is the type most frequently used for indicating transverse fatigue cracks in specimens whose length is great compared with their width. A typical example would be a crankshaft.

Circular Magnetisation—When an electric current is passed through a conductor, which may be of copper wire or bar, or the specimen itself undergoing examination, a circular field develops in and around the conductor, and if the latter is passed through the centre of a hollow object, such as a tube or a ring, a circular magnetic field will be induced in the hollow object. If current is passed through the part itself, the part serves as a conductor and the circular field is produced in it.

Principles involved in Magnetic Flaw Detection

We will first consider again the simplest form of magnet consisting of a straight bar and having a north pole at one end and a south pole at the other. If this bar is bent until its ends almost meet, as shown in Fig. 1*a*, the lines of force take new paths, and there is a close and powerful linkage between the opposite poles where the field is very concentrated. If the bending operation is continued until the two ends meet, and if they are welded together, a closed ring is formed (Fig. 1*b*). The magnetic field then has a completed iron path which it follows because the latter offers less reluctance (magnetic resistance) to the passage of a magnetic field than does air. The ring is said to be circularly magnetised, and there is no external polarity and very little detectable external magnetic field surrounding the ring.

If a slot is now sawn part of the way through the ring, as shown in Fig. 1*c*, an entirely new set of conditions is produced, and certain interesting changes which take place in the magnetic lines of force may be noted. They can be considered as following three different paths.



(a) (b) (c)

Fig. 1.—(a) Magnet field obtained by bending a bar magnet into a ring, leaving a small gap ; (b) if the bending is continued until the ends meet, and they are then welded together, the magnetic field follows the new path almost exclusively ; (c) when a slot is sawn in the ring an external leakage field is produced.

One of these is through the air and is called local leakage field. The second path passes from one edge of the gap through the small air space to the other edge, and the third passes through the remaining mass of the metal and is slightly distorted immediately under the gap. When a very small compass is brought up to the gap, it can be demonstrated that the north end of the compass needle is attracted by one side of the gap and repelled by the other. In other words the gap has a north pole on one side and a south pole on the other : a pair of poles having developed. Local leakage fields such as the one just described are produced by other abrupt discontinuities in the path of the flux, such as non-metallic inclusions, slag stringers, or cracks resulting from improper quenching of hardened steel.

As previously mentioned, when a conductor is carrying a current, there is a magnetic field surrounding it. It was also stated that if a tubular piece of magnetisable metal surrounded the conductor a circular field would be set up in the tube. This is a similar condition to that described above and it will be appreciated that a tube or cylinder may take the place of the bar magnet bent into a ring. Any abrupt discontinuity in the path of a magnetic field traversing the tube or cylinder would cause a local leakage field, as it would in the case of the ring.

When a conductor carrying a current is bent into a loop or series of loops, a solenoid is produced, and if a bar of magnetisable material is placed within the solenoid, the bar becomes magnetised and develops north and south poles at opposite ends. This type of magnetisation is called longitudinal, and together with circular magnetisation forms the two types used in crack detection.

We have seen that with magnetisable material it is possible to produce local leakage fields by longitudinal or circular magnetisation. The next step in crack detection is to locate these local leakage fields. This is done by introducing small particles of metal or metallic oxide of suitable magnetic characteristics into the vicinity of such fields. These particles are attracted to the leakage fields and hence tend to indicate their intensity and extent. In order to obtain the largest possible leakage field, it is important that the lines of magnetic force should cross the direction of the gap at as nearly a right angle as possible. For this reason transverse defects are sought in a bar by magnetising it longitudinally, and longitudinal defects are best discovered by magnetising the bar circularly.

The small magnetisable particles used for detecting the local leakage fields produced by cracks and other discontinuities may be in the form of dry powders or in the form of a suspension in a light oil. In the latter case, the liquid suspension may be flowed over the specimen or the specimen itself may be dipped in a container of the liquid. The particles are then attracted by the local leakage fields and held to the defect because of the local poles created on each side of it.

By the way of an example, consider a bar containing a transverse crack. In order to locate this defect the bar would be placed within a magnetising coil and a current passed through the coil. The field thus created would pass longitudinally through the bar. A north pole would be set up on one side of the crack and a south pole on the other, and the leakage field, from pole to pole, across the crack would attract and hold the magnetic powder when this was applied to the bar. The powder is attracted because the magnetic field exerts a force and attracts the particles to it which will conduct the field with less

Fig. 2. The magnetic field which exists when a round bar contains a small hole near the surface.

reluctance than air. In the case of a sub-surface defect and its effect on the field, Fig. 2 shows a cross-section through a round bar which has had a small hole drilled in one end near the surface. When a current is passed through the bar from end to end, a circular field is set up as shown by the dotted lines. The hole acts as a partial obstacle and the field diverges round it. Part of the field is crowded down into the bar under the hole and some is deflected between the hole and the surface. This deflection of the field forces some of the field outside the surface of the piece and this leakage field holds the powder. If the hole were at the centre of the bar the field would flow around it without interruption and there would be no leakage, nor would there be any indication from powder applied to the surface.

To summarise this section it can be stated that two main principles are used in magnetic crack detection :—

(1) Sudden breaks in the path of magnetic fields flowing through a specimen, such as might be caused by inclusions or any kind of crack, result in local flux leakage fields.

(2) When small particles having suitable magnetic properties are brought into the vicinity of local leakage fields, they are attracted to and held by these fields. The pattern formed gives a satisfactory picture of the shape and extent of the defects.

(To be continued)

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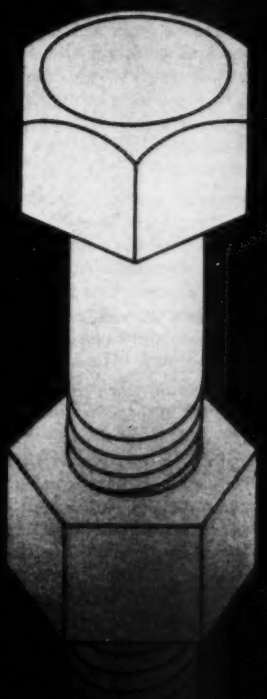

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Determination of Zinc in Aluminium and Copper Alloys

By M. Freegarde, B.Sc.

Bragg Laboratory, Naval Ordnance Inspection Department, Sheffield

An anion exchange method is described for the separation of zinc in the range 0·1–6% from all normal aluminium alloys and copper-base alloys not containing tin. The separated zinc is determined by titration with disodium ethylene diamine tetra-acetate (E.D.T.A.). The efficiencies of various eluting agents have been investigated, and the precision and accuracy of the method analysed.

THE separations obtained involve the formation and selective adsorbtion of complex metallic anions on the strongly basic anion exchange resin Deacidite FF. With hydrochloric acid, zinc and copper form various ions in solution of the type $MCl_n^{(n-2)-}$ the relative stabilities depending upon the exact conditions. In this case, the most important of these is MCl_4^{2-} both for copper and zinc, the latter being completely in the anionic form, the copper to a much smaller extent.

Maximum adsorbtion is obtained from 0·12 N hydrochloric acid containing 100 g./l. of sodium chloride,[1] under which conditions all the zinc and some copper are retained. No other metals occurring in aluminium alloys and bronzes have been found to be retained at all. It has been found that copper can be removed by washing with hydrochloric acid of from N to 2 N strength, the amount of zinc removed by this operation being a negligible minimum in 2 N solution. This is effected before elution of the zinc, for which purpose 0·005 N hydrochloric acid,[2] 1 N nitric acid, or 2 N caustic soda containing 20 g./l. of sodium chloride may be used. The last of these has been excluded, as the solution is then not suitable for titration with E.D.T.A. Of the other two methods, it has been found that 1 N nitric acid is to be preferred as eluting agent, since, although both methods are equally precise, a considerable saving in time results by using nitric acid.

Separation Method

Reagents

Solution R—0·12 N hydrochloric acid containing 100 g./l. sodium chloride.

Solvent Acid—100 ml. conc. hydrochloric acid + 100 ml. conc. nitric acid diluted to 400 ml. with water.

Nitric Acid—1 N.

Hydrochloric Acid—2 N.

Hydrochloric Acid—0·005 N.

Preparation of Column

The columns used are of 1 cm. internal diameter, and are about 25 cm. long, being fitted via a ground glass joint with a funnel. The resin as supplied (Deacidite FF, 16 to 50 mesh) is settled in 0·5 N hydrochloric acid for several minutes, after which the supernatant liquid is removed. The slurry remaining is poured into the column to give a depth of resin of about 15 cm. This is successively washed by 100 ml. of 1 N nitric acid and then 100 ml. of solution R. The column is now ready for use.

Procedure

A suitable weight of sample containing zinc up to 40 mg. is dissolved in 40 ml. of solvent acid, warming if necessary, and the solution taken to dryness. The solid is taken up in solution R and any insoluble material removed. This solution is run through the column at a flow rate of 6 ml./min. and is followed by 100 ml. of solution R, which washes the system free of aluminium, and 2 N hydrochloric acid, which removes any copper. Of this 20 ml. are sufficient for aluminium alloys and 100 ml. for copper-base alloys. The zinc is then eluted by passing 250 ml. of 1 N nitric acid and then titrated. The resin is regenerated by passing 30 ml. of solution R, and the only requirement for efficient running is that the resin should never be allowed to run dry.

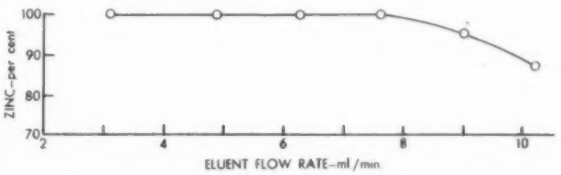

Fig. 1.—Percentage of zinc recovered from a column by a given volume of eluent at different flow rates.

Titration

Reagents

Buffer Solution—50 ml. of solution containing 8·8 ml. of 0·88 ammonia, and 10 g. ammonium chloride in 1 litre.

Indicator—0·05% solution in methylated spirits of 1-(2-pyridyl-azo) 2-naphthol.

E.D.T.A.—Aqueous solution, 1 ml. ≡ 1·63 mg. of zinc.

Copper Solution—Aqueous, 1 ml. ≡ 1 ml. of E.D.T.A. solution.

Procedure

The eluent is just made alkaline to litmus with ammonia solution (s.g. 0·88). After buffering, indicator is added, and E.D.T.A. solution run in to excess, the excess being determined by addition of a standard copper solution.

Discussion

Flow Rate

Since this method was designed for a routine laboratory, the optimum rate of flow for a column of given dimensions was of obvious importance. The maximum rate of flow for which complete exchange occurred was determined by varying the rate of elution of zinc from 3 to 10 ml./min. Since all operations on an ion exchange resin are basically similar, the same criticality of flow rate would be expected to hold in all cases. There were found to be no detectable differences in results up to a flow rate of about 7 ml./min., but above this figure low

TABLE I—RESULTS ON ROUTINE SAMPLES

Base Metal	Zinc Content (%)	
	Routine Methods	Ion Exchange Method
Aluminium	1·84	1·88, 1·80, 1·78
Aluminium	6·10	5·81, 5·76, 5·83, 6·01
Aluminium	3·53	3·59, 3·42, 3·44
Aluminium	0·10	0·095, 0·098
Aluminium	5·70	5·71, 5·68
Aluminium	5·64	5·68, 5·66
Aluminium	5·74	5·74, 5·70
Copper	1·15	1·23, 1·11, 1·10
Copper	0·10	0·11, 0·10
Copper	0·22	0·23, 0·22

results were obtained. (Fig. 1) This was to be expected, since exchange processes depend upon the setting up of equilibria at all exchange points, and this of necessity requires time, which a high flow rate does not allow. It was apparent therefore that a flow rate of 6 ml./min. was a safe working limit.

Eluting Agents

The results of Table II show that both eluting agents are equally acceptable, but 1 N nitric acid is to be preferred, since with it all the zinc is removed by passing 250 ml., while with 0·005 N hydrochloric acid, 500 ml. is required. The titration procedure applies equally well to both methods.

Exchange Kinetics

It was thought worthwhile to consider the possibilities of employing a knowledge of the kinetics of the exchange process to the purely utilitarian end of estimating the total amount of zinc on a column without eluting all of it. This procedure would be useful should a fairly good figure be required urgently. The elution stage consumes the larger part of the total time of the whole determination and this was considered in detail.

Using a steady flow rate at constant temperature, the

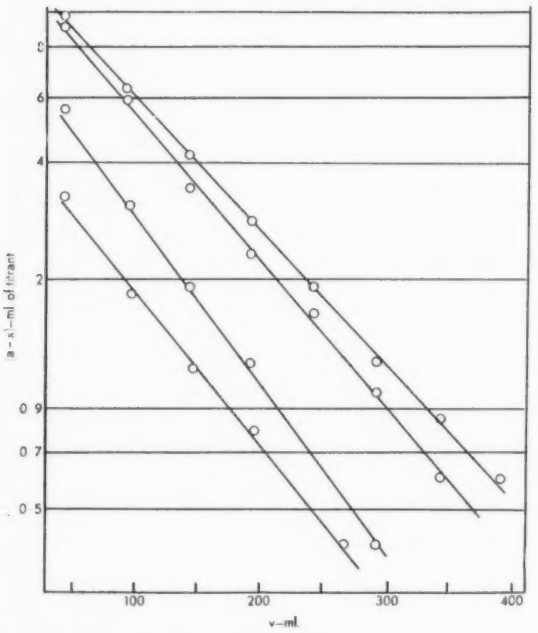

Fig. 2.—The relation between log (a—x), expressed as the equivalent amount of titrant, and v, the volume of eluent passed.

TABLE II.—COMPARISON OF METHODS USING B.C.S. ALUMINIUM ALLOY A

Method	Number of Determinations	Mean Value	Standard Deviation
Ion Exchange using 0·005N HCl	10	2·42	0·06
Ion Exchange using 1N HNO_3	10	2·41	0·05
Normal Chemical	10	2·42	0·18

TABLE III—COMPARISON OF FULL AND SHORTENED PROCEDURES

Sample	Zinc Content (%)		Difference (%)
	Using Full Procedure	Using Empirical Kinetic Relation	
1	3·44	3·46	0·6
2	1·78	1·77	0·6
3	5·76	5·81	1·0
4	2·40	2·46	2·5
5	0·140	0·137	2·1
6	1·01	1·06	2·0
7	5·70	5·78	1·4

rate of elution was investigated as a function of the volume of eluent passed. It was found, as Fig. 2 shows, that the exchange process obeys first order kinetics; that is, the rate of elution at any stage is proportional to the amount of ion remaining on the column at that time. If x is the amount of zinc eluted and v the volume of agent passed at that time, $dx/dv = -kx$ and $\log (a-x) = kv$, where a is the total amount of zinc initially absorbed, and k is a constant. Thus, it is a justified practical procedure to terminate the elution at any stage, to determine the amount of zinc in the measured volume of eluent collected, and hence to estimate the total amount of zinc from the pre-determined nature of the exchange. As an example, Table III shows a comparison between values obtained by this procedure and those obtained by the full procedure, the empirical relation being used that 95% of the total zinc on a column is eluted by the passage of 60% of that quantity of eluting agent which is required for complete elution. It can be seen that the values obtained are usually within 2% of the figure obtained by the full procedure. This method affords a saving in the total time of about 25%.

Range of Application

Results of Table I show the method to be satisfactory for zinc in the range 0·1–6% in alloys of copper and aluminium.

Interfering Elements

No noticeable interference has been observed due to the presence of magnesium, iron, manganese, nickel, titanium and silicon. Work has shown that in the case of alloys containing tin, it is essential to remove the tin by normal procedures before applying the ion exchange separation for zinc.

Accuracy and Precision

The precision and accuracy of the methods have been established by comparing the results of ten determinations, using 1 N nitric acid as eluting agent, upon B.C.S. Aluminium Alloy A, with ten determinations on the same alloy by classical methods. A statistical inspection shows that the ion exchange method furnishes results of equal accuracy but of superior precision.

Acknowledgment

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REFERENCES

1 Kallmann, Steele & Norton, *Anal. Chem.*, **23**, 11, 230.
2 Lower & Garney, *Anal. Chem.* **27**, XII, 1965.